UNIFORM FEDERAL POLICY

QUALITY ASSURANCE PROJECT PLAN

For

The Characterization of Lead in Soil at the Lead Sweetening Area and Ethyl Blending Area

WILCOX OIL SUPERFUND SITE

Bristow, Creek County, Oklahoma

September 22, 2017

Revision 1

Prepared for:

U.S. EPA Region 6

Dallas, Texas

Prepared by: Technology Integration and Information Branch (TIIB), Office of Superfund Remediation and Technology Innovation (OSRTI), Washington, DC 20460

and

ICF, 9300 Lee Highway, Fairfax, VA 22031 USA

Contract No.: EP-W-14-001; OSRTI Technical Assessment, Support, Outreach and Training

Technical Direction No.: Task Order TO 006/TD 013

QAPP Worksheet #1 & 2: Title and Approval Page

(UFP-QAPP Manual Section 2.1)
(EPA 2106-G-05 Section 2.2.1)

1. **Project Identifying Information**
    a. **Site name/project name:** Wilcox Oil Superfund Site, Characterization of Lead at Two Sites
    b. **Site location/number:** Bristow, Creek County, Oklahoma (06GG/0604942, OU 1)
    c. **Contract/Work assignment number:** EP-W-14-001/TO 006, Technical Directive 013

2. **Lead Organization:** EPA OSRTI TIIB

Prepared by: Deana Crumbing, EPA OSRTI TIIB

_____   _____
                                                                 Date

3. **Federal Regulatory Agency**

Approved by: Katrina Higgins-Coltrain, EPA Region 6, RPM and Project Lead

_____
EPA Region 6 Remedial Project Manager   Date

Approved by: David Charters, Quality Assurance Manager, EPA OSRTI

_____
EPA OSRTI Quality Assurance Manager   Date

4. **State Regulatory Agency:** Oklahoma Department of Environmental Quality (ODEQ) is an observer, assisting EPA with planning and implementing Long-term Sampling and Response Plan at Wilcox Oil. ODEQ is not a signatory for the QAPP.
5. **Other Stakeholders:** Not Applicable
6. **List plans and reports from previous investigations relevant to this project:** Not Applicable

Signatures indicate that officials have reviewed the QAPP and concur with its implementation it as written.
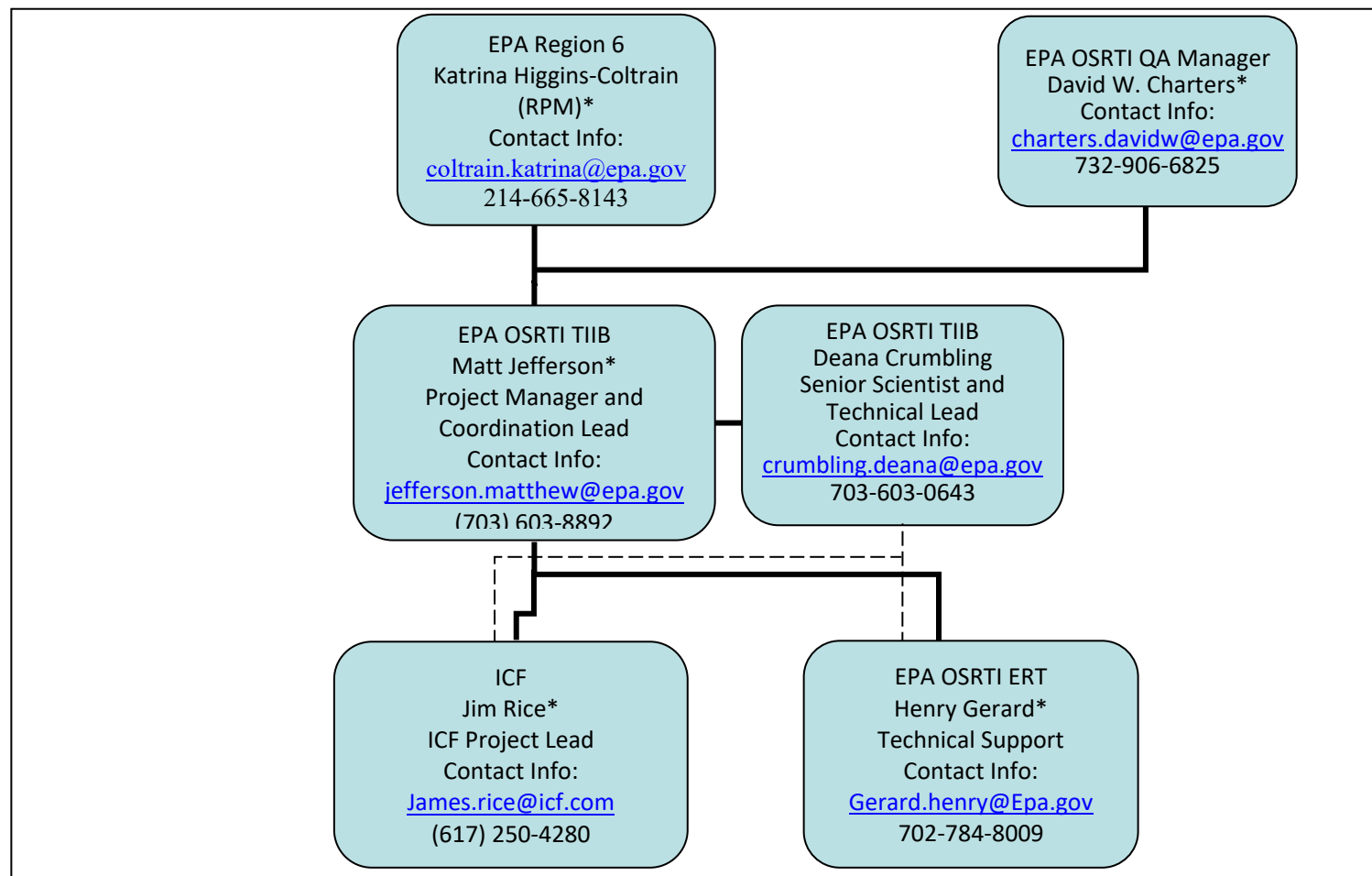
QAPP Worksheet #3 & 5: Project Organization and QAPP Distribution
(UFP-QAPP Manual Section 2.3 and 2.4)
(EPA 2106-G-05 Section 2.2.3 and 2.2.4)

The most current and approved copy of the QAPP will be delivered to recipients using email or a web-based system in use by EPA at the time of submittal.

*QAPP recipient   Lines of authority _____   Lines of Communication ---------------------------

EPA Region 6
Katrina Higgins-Coltrain
(RPM)*
Contact Info:
coltrain.katrina@epa.gov
214-665-8143

EPA OSRTI QA Manager
David W. Charters*
Contact Info:
charters.davidw@epa.gov
732-906-6825

EPA OSRTI TIIB
Matt Jefferson*
Project Manager and
Coordination Lead
Contact Info:
jefferson.matthew@epa.gov
(703) 603-8892

EPA OSRTI TIIB
Deana Crumbling
Senior Scientist and
Technical Lead
Contact Info:
crumbling.deana@epa.gov
703-603-0643

ICF
Jim Rice*
ICF Project Lead
Contact Info:
James.rice@icf.com
(617) 250-4280

EPA OSRTI ERT
Henry Gerard*
Technical Support
Contact Info:
Gerard.henry@Epa.gov
702-784-8009

QAPP Worksheet #4, 7 & 8: Personnel Qualifications and Sign-off Sheet
(UFP-QAPP Manual Sections 2.3.2 – 2.3.4)
(EPA 2106-G-05 Section 2.2.1 and 2.2.7)

ORGANIZATION: EPA OSRTI

Name Signature/Date	Project Title/Role	Education/Experience	Specialized Training/Certifications
Matt Jefferson	**HQ Coordination Lead, Project Manager for Study.** Responsible for providing management and technical oversight during all field, laboratory, and analysis activities. Coordinates with EPA Region 6 RPM to ensure project goals are being met. Review and sign-off on QAPPs and any future modifications to the plans; provides quality-related direction to the Project QA Manager; and has authority to suspend affected project or lab activities if approved quality requirements are not adequately met. Operational support as needed.	BS, MS Environmental Engineering, 20 years RPM experience in several regions	EPA TOCOR, XRF Radiation Safety, XRF operation, 40-hour HAZWOPER
Deana Crumbling	**Technical Lead, Senior Scientist.** Assist with study design and QAPP preparation; sample processing and analysis team lead; technical and operational support to sample prep and field teams, Niton instrument analysis leader, lead evaluator of the study data using statistics and any other suitable tools; conduit of experimental results to other EPA parties interested in XRF application to risk assessment and site cleanups.	BS, biochemistry; MS, environmental science; 20+ years as analytical chemist	XRF Radiation Safety, 40-hour HAZWOPER

ORGANIZATION: EPA Region 8 Superfund

Name Signature/Date	Project Title/Role	Education/Experience	Specialized Training/Certifications
Katrina Higgins-Coltrain	**EPA Region 6 RPM for the Wilcox Oil Site.** Project proponent with overall responsibility for the lead delineation project to support project specific needs (Remedial Investigation). Approves all project level decisions and coordinates Region 6 contractor and OSRTI actions.	MS Geology, 20 years as an RPM	EPA TOM, 40-hour HAZWOPER
EPA Region 6 START Contractor support staff	**Field and Logistics Support**. START contractor may be on site conducting other work during part of the lead delineation and available to help support general field logistics. As required for task.	various	Field health and safety training

ORGANIZATION: ICF (Contractor to OSRTI)

Name Signature/Date	Project Title/Role	Education/Experience	Specialized Training/Certifications
Jim Rice	**Field Sampling Team Lead and Technical Support.** Assists with preparation of QAPP and SOPs, Field Sample Team Lead; technical support to Sample Prep and XRF instrument analysis team. Provides initial level of quality assurance through adherence to the QAPP, self-check, and good laboratory practice; liaison with off-site geostatistical analysis staff	BS, MS Geology, 25 years conducting site investigations and supporting technology analysis and transfer. Field QA officer under EPA RAC, ARCS and other agency cleanup contracts.	XRF Radiation Safety, 40-Hour OSHA Hazardous Waste Site Worker Training; OSHA Supervisor Training, 8 Hour OSHA Refresher Training; First Aid and CPR
Paul Zarella	**Technical Support.** Provides support for preparation of QAPP and SOPs, oversees field activities, conducts XRF analysis and supports the project as needed in field or lab.	BA, MSc Geology, 3 years planning and implementing site investigations, GIS and chemical data analysis.	XRF Radiation Safety, 40-Hour OSHA Hazardous Waste Site Worker Training; 8 Hour OSHA Refresher Training;

ORGANIZATION: EPA OSRTI ERT

Name Signature/Date	Project Title/Role	Education/Experience	Specialized Training/Certifications
Henry Gerard	**Technical Support, Health and Safety.** Technical and field support for sample collection, sample processing and XRF analysis using ERT's Olympus XRF. Provide initial level of quality assurance through adherence to the QAPP, self-check, and good laboratory practice. POC for all field safety issues.	Degrees in Chemistry and Biology, extensive experience in field analytical implementation	XRF Radiation Safety, lead for the USEPA ERTG-XRF Subgroup.
Other ERT support staff and/or SERAS Contractor support	**Technical Support.** Additional staff to be assigned to the project as needed to support field sample collection, sample processing, and XRF analysis.	various	As required for task. Any staff operating the XRF will have taken the XRF Radiation Safety Training course.
David W. Charters	EPA OSRTI Quality Assurance Manager. Review/approve QAPP prepared by OSRTI TIIB		

*Signatures indicate personnel have read and agree to implement this QAPP as written

QAPP Worksheet #6: Communication Pathways
(UFP-QAPP Manual Section 2.4.2)
(EPA 2106-G-05 Section 2.2.4)

Mobile phone numbers are included here for all EPA OSRTI TIIB staff because most of the communication needs will be in the field.

Communication Driver	Organization	Name	Contact Information	Procedure (timing, pathway, documentation, etc.)
Project Objectives, Site access and Logistics	EPA Region 6 Remedial Project Manager	Katrina Higgins-Coltrain	214-665-8143	As the proponent of the study RPM will communicate project needs to the OSRTI TIIB team. Coordinates with START contractor and site contacts to support logistics for TIIB team.
Quality Management/Quality Assurance Officer	EPA OSRTI	David W. Charters	732-906-6825	QAO will remain independent of direct project involvement and day-to-day operations. The QAO will ensure implementation of the quality assurance elements outlined in this QAPP. The QAO will be the point of contact with the Project Manager for quality matters.
Manage all Project Phases	EPA OSRTI TIIB	Matt Jefferson	703 209-4784	Project manager will manage project personnel, and serve as liaison to the Region 6 team. Manage day to day operations of the project. Reports to Region 6 RPM issues with cost, schedule, etc.

Coordination and communication of fieldwork activities	ICF	Field Sampling Team leader Jim Rice	978 590-5852	Field team leader will communicate relevant field information to the project manager, technical lead, and team members.
Field data and quality control reports				Field team leader will generate and report data and documents as required by this UFP QAPP along with quality control reports to the Site project manager.
Coordination of sampling supplies for field activities				The Field Team Leader will ensure all sample containers and appropriate shipping materials (such as coolers and bags) are mobilized before field sampling begins and throughout the project.
Internal chain-of-custody records and sampling documentation				Internal chain-of-custody records and sampling documentation will be submitted to the field lab at the end of each day that samples are collected.
Minor deviations from QAPP procedures identified during field activities	ICF	Field Sampling Team leader Jim Rice	978 590-5852	The Field Team Leader will verbally request a field change for any minor changes in sampling procedures that occur as a result of conditions in the field. Approval from Tech Lead is required before the change is initiated. This request will be documented in the field log book.

QAPP amendments	EPA OSRTI TIIB	Technical Lead Deana Crumbling	571 215-6947	Any changes to the QAPP prior to field work will require the Technical Lead to prepare an addendum that will be approved by the PM and EPA RPM before any changes are implemented.
QAPP – routine communications regarding analyses during implementation	EPA OSRTI TIIB	Technical Lead Deana Crumbling	571 215-6947	Primary point of contact to ensure that field analytical activities comply with the QAPP so that resulting data will meet data quality objectives.
QAPP changes during project execution (sample prep and analysis)	EPA OSRTI TIIB	Technical Lead Deana Crumbling	crumbling.deana@epa.gov 571 215-6947	Communicates the need for, and content of revisions to RPM and Region 8 6 RPM for approval. Advises project teams of changes.
Data verification issues, e.g., incomplete records	EPA OSRTI TIIB	Technical Lead Deana Crumbling	crumbling.deana@epa.gov 571 215-6947	Communicates issues to RPM and Quality Manager for resolution
Data review corrective actions	EPA OSRTI TIIB	Technical Lead Deana Crumbling	crumbling.deana@epa.gov 571 215-6947	Communicates issues to RPM and Quality Manager for resolution

QAPP Worksheet #9a: Project Planning Session Summary
(UFP-QAPP Manual Section 2.5.1 and Figures 9-12)
(EPA 2106-G-05 Section 2.2.5)

Project Name: Characterization of Lead in Soil at the Lead Sweetening Area and Ethyl Blending Area, Wilcox Oil Superfund Site **Project Manager:** Katrina Higgins-Coltrain	**Site Name:** Wilcox Oil **Site Location:** Bristow, OK

Date of Session: Monday, June 3, 2016
Systematic Planning Meeting Purpose: Conference Call scoping meeting for all areas of Wilcox Site (included discussion of EBA and LSA)

Name	Title	Affiliation	Phone #	E-mail Address
Katrina Higgins-Coltrain	Remedial Project Manager	EPA R6	214-665-8143	coltrain.katrina@epa.gov
Phil Turner	Risk Assessor	EPA R6	214-665-2706	turner.philip@epa.gov
Teri Mcmillan	Project Manager	EA Engineering	505 224-9013	tmcmillan@eaest.com
Tom Kady	ERT	EPA ERT	732-735-5822	Kady.Thomas@epa.gov
Matthew Jefferson	Environmental Engineer	EPA TIIB	703-603-8892	jefferson.matthew@epa.gov
Deana Crumbling	Staff scientist	EPA TIIB	703-603-0643	Crumbling.Deana@epa.gov
Ed Gilbert	Hydrogeologist	EPA TIIB	703-603-8883	Gilbert.edward@Epa.gov
Jim Rice	Geologist	ICF	617-250-4280	James.Rice@icf.com
Todd Downham	Project Manager	Oklahoma DEQ	405-702-5136	Todd.Downham@deq.ok.gov
Barry Forsyth	EPA Liaison	USFWS	214 665 8467	Forsythe.Barry@epa.gov

Key Decisions: EBA and LSA will be part of Phase 1 (Mobilization 1) delineation. EPA OSRTI TIIB Field Team will utilize XRF (30-point incremental composite soil sampling and other techniques) to identify the areas where Pb concentrations in soil (0 to 24 inches) exceed 200 ppm, the preliminary remediation goal.
Action Items: OSRTI will prepare a work plan and QAPP for the Pb delineation.

QAPP Worksheet #9b
Systematic Planning Meeting Participants Sheet

<table>
<tr>
<td>

Project Name: Characterization of Lead in Soil at the Lead Sweetening Area and Ethyl Blending Area, Wilcox Oil Superfund Site

Project Manager: Katrina Higgins-Coltrain
</td>
<td>

Site Name: Wilcox Oil

Site Location: Bristow, OK
</td>
</tr>
</table>

Date of Session: Thursday July 14, 2016
Systematic Planning Meeting Purpose: In-person meeting to develop sampling strategy

Name	Title	Affiliation	Phone #	E-mail Address
Matthew Jefferson	Environmental Engineer	EPA Superfund TIIB	703-603-8892	jefferson.matthew@epa.gov
Deana Crumbling	Staff scientist	EPA Superfund TIIB	703-603-0643	Crumbling.Deana@epa.gov
Jim Rice	Geologist	ICF	617-250-4280	James.Rice@icf.com

Key Decisions: Decision is made to sample along transects using sampling units (SUs) of 9 to16 points within 4 to 9 square feet, and then develop definitive DUs to confirm < 200 ppm. Recon and SU sampling at EBA to identify potential source areas prior to DU sampling. Sampling will require developing an equipment list.
Action Items: OSRTI will begin developing SFP, Decision Logic Diagrams and QAPP. Develop sampling equipment list.

QAPP Worksheet #9c
Systematic Planning Meeting Participants Sheet

<table>
<tr><td colspan="2">Project Name: Characterization of Lead in Soil at the Lead Sweetening Area and Ethyl Blending Area, Wilcox Oil Superfund Site

Project Manager: Katrina Higgins-Coltrain</td><td colspan="3">Site Name: Wilcox Oil

Site Location: Bristow, OK</td></tr>
<tr><td colspan="5">Date of Session: Wednesday, August 24, 2016
Systematic Planning Meeting Purpose: Field reconnaissance to observe site conditions.</td></tr>
<tr><td>Name</td><td>Title</td><td>Affiliation</td><td>Phone #</td><td>E-mail Address</td></tr>
<tr><td>Matthew Jefferson</td><td>Environmental Engineer</td><td>EPA Superfund TIIB</td><td>(703) 603-8892</td><td>jefferson.matthew@epa.gov</td></tr>
<tr><td>Deana Crumbling</td><td>Staff scientist</td><td>EPA Superfund TIIB</td><td>703-603-0643</td><td>Crumbling.Deana@epa.gov</td></tr>
<tr><td colspan="5">Key Decisions: Identified additional potential source area and some logistical concerns including a number of fences that prevent access along the west side of the EBA and east and southern boundaries of the LSA – investigation will not end at fences, unless access prevents it. Two buildings are present at EBA, on with dirt floor - we will sample the floor of the eastern building
Action Items: Incorporate these considerations into the FSP and QAPP.</td></tr>
</table>

QAPP Worksheet #9d
Systematic Planning Meeting Participants Sheet

Project Name: Characterization of Lead in Soil at the Lead Sweetening Area and Ethyl Blending Area, Wilcox Oil Superfund Site **Project Manager:** Karina Higgins-Coltrain	**Site Name:** Wilcox Oil **Site Location:** Bristow, OK

Dates of Discussion: August 14 - 18, 2017
Purpose of Discussions: The R6 RPM raised concerns that there may be a disruption of the federal government October 1, 2017, potentially interrupting the Pb characterization field work deployment. Consultation among the parties required to evaluate change in schedule.

Name	Title	Affiliation	Phone #	E-mail Address
Katrina Higgins-Coltrain	Remedial Project Manager	EPA R6	214-665-8143	coltrain.katrina@epa.gov
Matthew Jefferson	Environmental Engineer	EPA TIIB	703-603-8892	jefferson.matthew@epa.gov
Henry Girard	ERT	EPA ERT	702-784-8009	Gerard.henry@epa.gov
Deana Crumbling	Technical Lead, Senior Scientist	EPA TIIB	703-603-0643	Crumbling.Deana@epa.gov
Dan Powell	Chief, Technology Integration & Information Branch	EPA TIIB	703-603-7196	Powell.dan@epa.gov
Jim Rice	Geologist	ICF	617-250-4280	James.Rice@icf.com

Key Decisions: TIIB's Wilcox field effort would be moved from Sept 17 – Oct 7 to Oct 22 to Nov 11, 2017.

Action Items: Deana to finish QAPP revisions. David Charters (OSRTI/ERT QA Manager) will serve as the QAPP reviewer and approver.

QAPP Worksheet #9e (Placeholder)
Systematic Planning Meeting Participants Sheet

<table>
<tr><td colspan="2">Project Name: Characterization of Lead in Soil at the Lead Sweetening Area and Ethyl Blending Area, Wilcox Oil Superfund Site

Project Manager: Karina Higgins-Coltrain</td><td colspan="3">Site Name: Wilcox Oil

Site Location: Bristow, OK</td></tr>
<tr><td colspan="5">Date of Session: TBD
Systematic Planning Meeting Purpose: Systematic Planning meeting to review CSM for EBA and LSA and identify path to closure of these sites. Proposed attendees below</td></tr>
<tr><td>Name</td><td>Title</td><td>Affiliation</td><td>Phone #</td><td>E-mail Address</td></tr>
<tr><td>Katrina Higgins-Coltrain</td><td>Remedial Project Manager</td><td>EPA R6</td><td>214-665-8143</td><td>coltrain.katrina@epa.gov</td></tr>
<tr><td>Phil Turner</td><td>Risk Assessor</td><td>EPA R6</td><td>214-665-2706</td><td>turner.philip@epa.gov</td></tr>
<tr><td>Teri Mcmillan</td><td>Project Manager</td><td>EA Engineering</td><td>505 224-9013</td><td>tmcmillan@eaest.com</td></tr>
<tr><td>Tom Kady</td><td>ERT</td><td>EPA ERT</td><td>732-735-5822</td><td>Kady.Thomas@epa.gov</td></tr>
<tr><td>Matthew Jefferson</td><td>Environmental Engineer</td><td>EPA Superfund TIIB</td><td>703-603-8892</td><td>jefferson.matthew@epa.gov</td></tr>
<tr><td>Henry Girard</td><td>ERT</td><td>EPA ERT</td><td>702-784-8009</td><td>Gerard.henry@Epa.gov</td></tr>
<tr><td>Deana Crumbling</td><td>Staff scientist</td><td>EPA Superfund TIIB</td><td>703-603-0643</td><td>Crumbling.Deana@epa.gov</td></tr>
<tr><td>Jim Rice</td><td>Geologist</td><td>ICF</td><td>617-250-4280</td><td>James.Rice@icf.com</td></tr>
<tr><td>Todd Downham</td><td>Project Manager</td><td>Oklahoma DEQ</td><td>(O) 405-702-5136</td><td>Todd.Downham@deq.ok.gov</td></tr>
<tr><td>Barry Forsyth</td><td>EPA Liason</td><td>USFWS</td><td>214 665 8467</td><td>Forsythe.Barry@epa.gov</td></tr>
<tr><td colspan="5">Key Decisions: TBD
Action Items: TBD</td></tr>
</table>

QAPP Worksheet #10: Conceptual Site Model
(UFP-QAPP Manual Section 2.5.2)
(EPA 2106-G-05 Section 2.2.5)

Conceptual Site Model (CSM) for the Areas to be sampled during the Study

Wilcox Oil Company is an inactive and abandoned oil refinery located in Creek County, Oklahoma in the northeast section of the town of Bristow, Oklahoma (Figures 10-1 and 10-2).
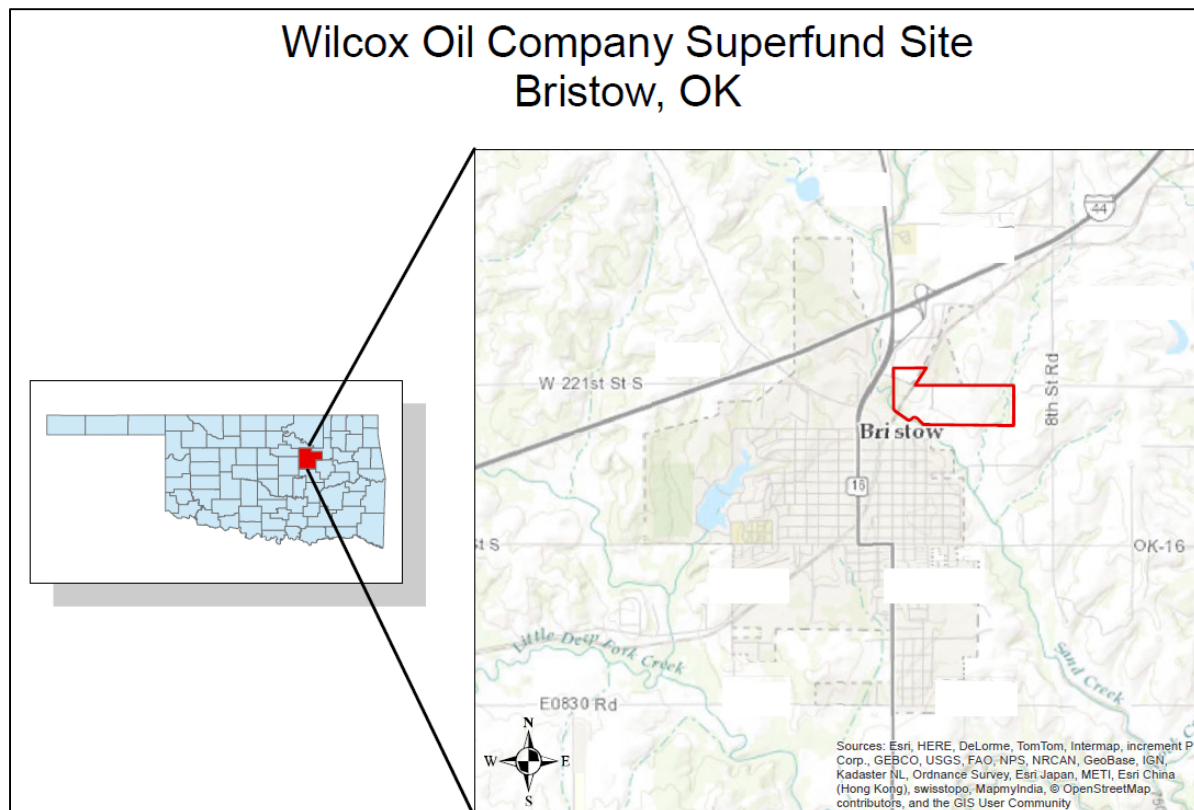

Figure 10-1.   Location of Wilcox Oil Site.

The site encompasses approximately 140 to 150 acres and includes five areas: the Wilcox Process Area, the Loraine Process Area, the Loading Dock Area, the North Tank Farm, and the East Tank Farm. Several preliminary investigations have occurred at the site beginning in 1994. In 2015, Lockheed Martin assisted the Environmental Protection Agency/ Environmental Response Team (EPA/ERT) and EPA Region 6 to perform a direct sensing investigation to qualitatively address the nature and extent of contamination at several sites using direct sensing tools including the rapid optical screening tool (ROST) and X-Ray fluorescence (XRF). Details of the site history and previous investigations can be found in the reports from these investigations (Lockheed Martin, 2016).

Within the Wilcox Process Area, the two areas containing lead as the contaminant of potential concern (COC) in soil include:
•   Lead Sweetening Area (LSA)
•   Ethyl Blending Area (EBA)
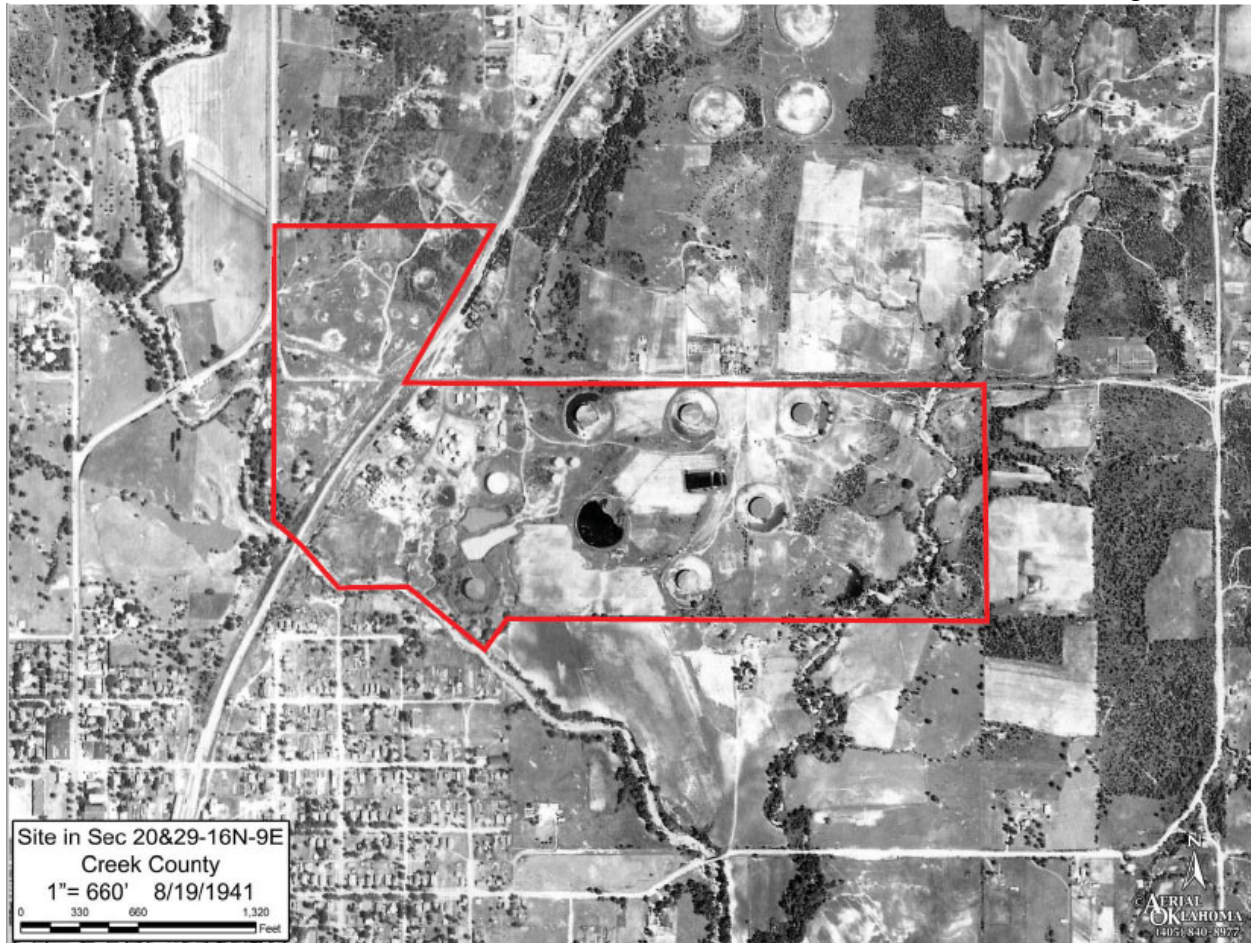
Figure 10-2.   1941 aerial photo of refinery footprint.

The **Lead Sweetening Area (LSA)** is located in the central portion of the Wilcox property. Historical maps indicate the area contained acid tanks, agitators, treaters, nearby "run down" tanks, and condensate tanks. The area was also called the "Doctor Process Area" (Figure 10-3).

The ERT report (Lockheed Martin, 2016) states that the chemicals used in the doctor sweetening process included sodium plumbite (Na_2PbO_2), sodium hydroxide (NaOH), and elemental sulfur or sulfonic acid to produce lead sulfide precipitate. The lead sulfide adsorbs mercaptan compounds that darken gasoline and give an offensive odor (Lachman, 1931). The ERT investigation identified phenolic compounds in the subsurface soils which were associated with the process. Crystals were observed on the surface in this area and XRF analysis showed that they contained very high lead content (percent levels above the calibration range). Vertical contamination of lead was investigated at five boring locations using single *in situ* XRF readings. In four of the locations, lead was found above 200 ppm in the upper foot of soil only, and at the remaining location it was detected at a maximum depth of 4 feet. Groundwater samples from the area show elevated lead concentrations (up to 151,000 mg/L - near Tank 34), and are believed to reflect perched water zones.

The LSA is currently very sandy with minimal plant growth and contains areas of sparkling sands/salts. Erosion is noted from this area to the south toward Sand Creek, and the tributary to Sand Creek to the east. Areas of dense trees, shrubs, poison ivy, and tall grasses surround the open sandy area. A metal fence extends through the eastern side of the investigation area, and there is a nearby elongated pond.
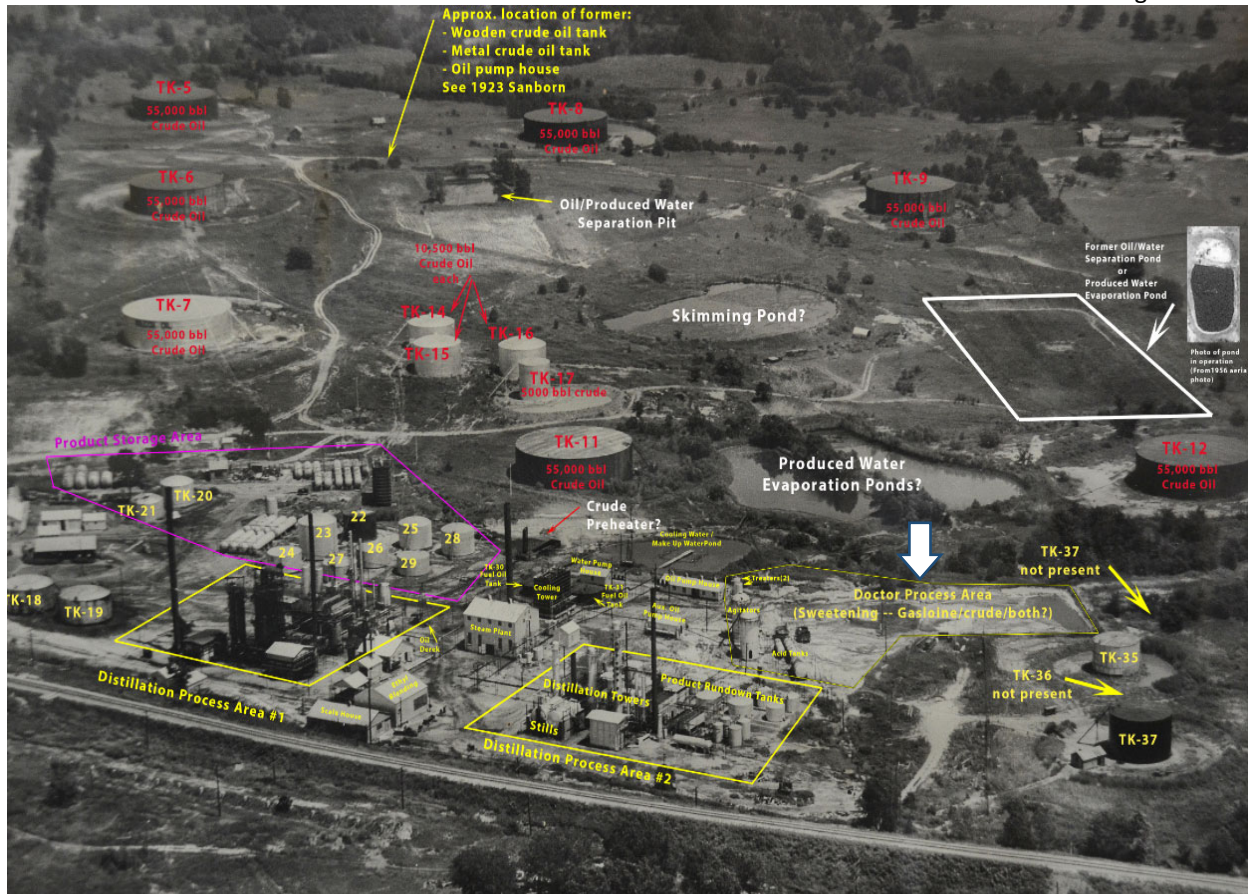
Figure 10-3.   Lead Sweetening Area (large white arrow).

Much of the infrastructure has been removed in the intervening years, and unmanaged vegetation has grown up throughout the area (Figure 10-4).

Modeling of the 2015 XRF data produced a contour map of lead concentration (Figure 10-5). The estimated area of concentration greater than 200 ppm is approximately 6 acres (600 feet north-south and 500 feet east-west) and the high concentration area, greater than 10,000 ppm, is approximately 0.6 acres (approximately 250 feet north-south and 100 feet east-west). Most of the soil lead measurements were taken with a handheld XRF at the ground surface, and there is limited data regarding the vertical extent of lead contamination. The subsurface sample near the center of the area analyzed showed high concentrations (greater than 5,000 ppm at a depth of 1 foot).

Figure 10-4. 2017 Google Earth image of the Lead Sweetening Area (LSA, yellow pin), and the Ethyl Blending Area structures (white arrow) by railroad track.
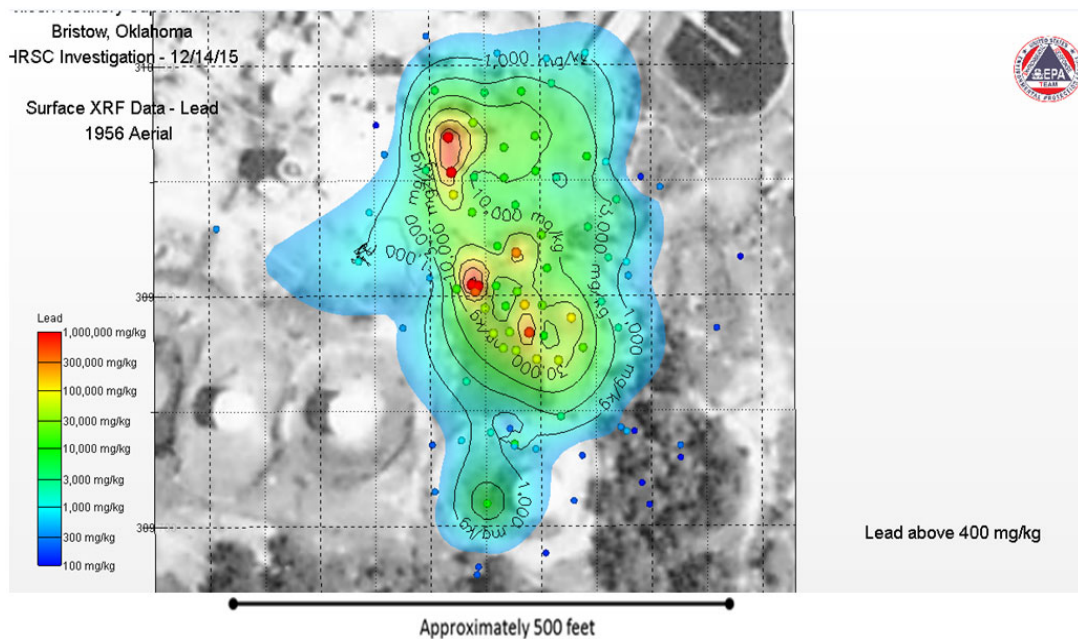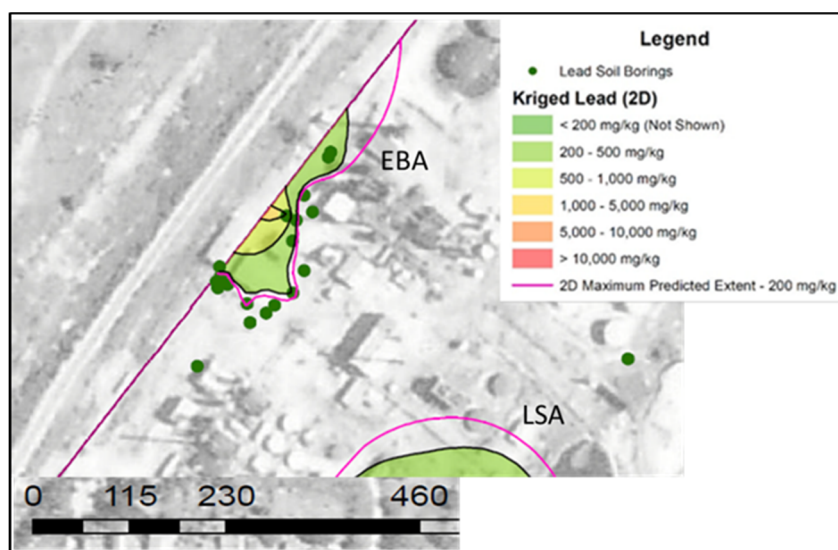

Figure 10-5. Geostatistical kriging iso-concentration lines produced from 2015 *in situ* XRF readings in the LSA (Kady, 2015).

The **Ethyl Blending Area (EBA)** is located in the Upper Wilcox Process Area in the northwest corner of the Wilcox Refinery. The area currently contains an intact main building measuring approximately 45 by 40 feet, with a sign reading "Ethyl Blending" and was presumably used to blend liquid tetraethyl lead (TEL) into the final gasoline product. TEL, which was TEL blended with the lead scavengers 1,2-dibromoethane and 1,2-dichloroethane, is normally supplied as ethyl fluid and contained a reddish dye to distinguish treated from untreated gasoline. Historical maps and photographs show no other permanent buildings within approximately 100 feet, but a rectangular feature (possible fence, slab or foundation) appears to be present on the southwest side of the building. Recent aerial photographs indicate the area is relatively flat and currently contains several large trees.

Historical aerial photographs show that between 1941 and 1956 the building was expanded to the northwest and the addition appears to have a lower roofline possibly indicating a storage area approximately 20 by 50 feet. Lighter colored ground on the southeast side of the building indicates surface disturbance. Access to the building includes a road parallel to the railroad and fence line that terminates at the northwest side (rear) of the building, and a larger road to the east that services several buildings in the area. A site visit by EPA staff in August 2016 found the area densely grown up with brush and poison ivy. Two structures are present. The main building is solidly enclosed with a concrete floor; one side of the building is very close to the railroad tracks. The adjacent structure is little more than a shed with open sides and with a dirt floor under which trucks may have stopped to load/unload. Rusted drums and fragments of equipment are present in and around the buildings. Small bore steel piping is still visible along sides of the buildings. There is at least one concrete basin that holds rain water.

The ERT investigation included screening surface soils with XRF at approximately 25 locations in the EBA. There is limited discussion of the EBA results in the SERAS report, but a contour map of lead concentration was prepared (Figure 10-6).

Figure 10-6.   Geostatistical kriging iso-concentration lines from 2015 *in situ* XRF readings around the EBA structures.

The map shows a possible area of lead contamination above the 200 ppm screening level near the building along the railroad track. The area greater than 200 ppm extends less than 100 feet to the east, but appears to be elongated several hundred feet in the north-south direction. Most readings were between 100 and 600 ppm. The maximum concentration is 1,449 ppm.

The problem to be addressed by the project: Region 6 is conducting a remedial investigation at the site in several phases (mobilizations) and has prioritized the site investigations based on potential risk.

Surface and near surface soils with lead concentration of greater than 200 ppm present a potential risk for direct contact and soil migration, and are priority sites. The RPM would like to identify the boundaries where lead exceeds 200 ppm in soils at both of these sites. There are two potential exposure zones to be delineated, 0 to 6 inches (human exposure) and 0 to 24 inches (ecological exposure). Deeper soils (greater than 2 feet) will not be investigated in this task, but one of the outcomes will be to identify potential areas where deeper soil investigation may be needed. The results of this study will be used to plan the next steps which may include risk management or removal actions.

QAPP Worksheet #11: Project/Data Quality Objectives
(UFP-QAPP Manual Section 2.6.1)
(EPA 2106-G-05 Section 2.2.6)

Statement of the Problem

Metals contamination related to industrial activities affects many areas in the U.S. Traditional soil and sediment investigations for metal contamination have relied on the collection of discrete samples, followed by analysis of grab subsamples in a distant laboratory. However, this strategy generally produces data sets that contain high degrees of data variability due to soil heterogeneity at the spatial scales of sample collection (100-300 grams) and metals analysis (0.5 to 1 gram). Since risk and cleanup decisions are made at much larger spatial scales (10s to 100s tons), statistical evaluations of soil data are needed to manage the chance of decision errors in risk and cleanup decisions. As a consequence of the high degree of data variability, very large numbers of discrete samples are required, incurring high costs and long time frames.

Incremental sampling is an alternative technique for collecting soil and sediment samples for contaminant analysis. Although new to remediation practitioners, incremental sampling has long been practiced in the mining and agricultural industries using the theory and practices outlined by Pierre Gy (USEPA 1999). Incremental sampling uses protocols for sample collection and sample processing that control soil heterogeneity so that fewer samples are required to produce statistically strong data sets. When used in conjunction with real-time sample processing and analysis, incremental sampling can reduce the costs and time frames involved in site investigation, while simultaneously producing high quality data that can support transparent and defensible decisions. This strategy has already proven its feasibility for several residential Pb sites, where incremental sampling has been paired with quality-controlled XRF analysis. This study will evaluate whether real-time XRF can be paired with 1) composite sampling of small square areas (termed sampling units, SUs) that function as sampling "points" on transects for the purpose of identifying concentration trends and locating boundaries; and 2) incremental sampling of larger-area decision units (DUs) that provide more accurate data to confirm the tentative location of excavation boundaries established by the SUs.

Historic operations at the Wilcox and Lorraine Oil refineries resulted in the release of hazardous materials to soil, sediment, surface water and groundwater. EPA Region 6 is conducting a remedial investigation to determine the nature and extent of the releases and if these releases present an unacceptable risk to human or environmental receptors. Preliminary soil screening at two of these sites, the Lead Sweetening Area (LSA) and Ethylbenzene Blending Area (EBA), identified the potential for lead at concentrations above the screening level of 200 ppm. EPA Region 6, along with cooperating agencies Oklahoma Department of Environmental Quality and US Fish and Wildlife Service, seek to identify areas where lead contamination in soil is greater than the 200 ppm screening level.

Goals of the Study and Information to be Generated

The overall goal of the study is to support the remedial investigation at the Wilcox oil site by characterizing lead concentrations at two locations. Specifically, the goals are:

1.   Identify the spatial extent of lead with a concentration of greater than 200 ppm in the 0 to 6 inch interval at the EBA and LSA for risk assessment and mitigation decisions. Estimate contaminated soil volumes for possible removal.

2.	Identify the spatial extent of lead with a concentration of greater than 200 ppm in the 0 to 24-inch interval at the EBA and LSA for risk assessment and mitigation decisions. Estimate contaminated soil volumes for possible removal.

3.	Introduce regional EPA staff to sampling, processing and analysis techniques for Incremental Composite Sampling and XRF to rapidly and definitively identify the concentration of lead in soil. Continual improvement and procedure development for Incremental Composite Sampling and XRF analysis are important aspects of this work for OSRTI TIIB. Field methods, sample preparation procedures and analytical procedures, will be evaluated as part of the study.

4.	Evaluate the relative distribution of Pb among soil particle size fractions for a few select samples. Determine whether higher Pb concentrations in finer particles are more likely to contribute to storm run-off to streams. Evaluate the particle size and Pb content of any crystalline material observed in the LSA to determine whether soil sieving at 100-mesh could remove this material and bias soil results. Retain samples of any crystalline material for potential laboratory evaluation of water solubility and/or other relevant characteristics.

Boundaries of this Study
1.	This study addresses the conditions at two specific areas within the Wilcox Oil site; the LSA and the EBA. Since this is a characterization effort, the boundaries of the two contaminated areas are only generally understood at this time. Characterization activities will cross fences and other obstacles to establish the 200 ppm boundaries.

2.	The data needed to support the decisions for this project will consist of XRF concentrations for Pb in two depth intervals: surface (0 to 6" depth interval) and shallow subsurface (6 to 24"). Although the data for the two depth intervals will be generated separately, the concentration of the entire 0 to 2 ft. interval can be generated mathematically if needed for risk assessment purposes.

3.	The only target analyte is Pb, although data for concentrations of other metals will be automatically collected by the XRF. Pb concentrations are expected to range from <50 ppm to >300,000 ppm. The XRFs have demonstrated linearity at least to 5600 ppm. XRF linearity and accuracy are unlikely for concentrations greater than 10,000 ppm, however, accurate reporting at such high concentrations is not necessary to meet the goals of the study.

4.	The process of establishing the 200-ppm Pb boundary will use soil samples sieved to 10-mesh (<2 mm particle size) to expedite sample processing. Once located, confirmation of the 200-ppm boundary will use the <100-mesh (<150 µm) soil particle size fraction, which is expected to be the most relevant particle size for risk evaluation, per the Superfund guidance for Pb-in-soil exposure (USEPA OLEM Directive 9200.1-128).

Rigor of the Sampling and Analytic Approach
The primary purpose of this study is to determine the spatial extent of lead soil contamination for two receptor groups (human and ecological). Soil that presents excessive risk will require some form of mitigation. Therefore, the data must have sufficient value to potential data users (RPMs, risk assessors, stakeholders, Superfund program managers, legal counsel, construction design etc.) to support confident decisions about site risk and optimal cleanup design. EPA policies regarding scientific integrity and data quality overwhelmingly favor sampling designs that are based on sound scientific and statistical principles, that are transparent and defensible, have undergone performance testing and peer review,

and that have error rates that are known to be acceptably low. Incremental-composite sampling (ICS) designs meet these requirements. When coupled with real-time, quality-controlled X-ray fluorescence (XRF) analysis, high density definitive Pb data can be generated rapidly and for much less cost than traditional laboratory analysis.

Reported concentrations for analytes in soil are considered definitive when the following conditions are met (USEPA 2006):

- The volume of soil over which the reported concentration value(s) apply (i.e., sample representativeness) are known, AND
- The uncertainty range around a data point (concentration), or statistics derived from a data set (such as a mean and UCL) are known, AND
- The identity of the target analyte is known.

ICS/XRF designs can rapidly generate high quality, representative, definitive data and minimize data gaps in a single mobilization using real-time adaptive decision-making. In comparison to traditional static, discrete sample/fixed lab analysis designs, ICS/XRF for many metal analytes (such as lead, arsenic and mercury) can provide higher confidence to decision-makers and stakeholders that characterization is complete and optimal mitigation options can be selected and implemented. These benefits outweigh the cost of more highly trained staff and more sophisticated procedures needed to design and implement ICS/XRF field efforts. The benefits to the project's bottom line include:

- a statistically superior and more reliable reported data result (since XRF can rapidly perform multiple analyses from the same sample at no extra cost, in contrast to the single analysis performed by traditional laboratories);
- fewer field mobilization cycles to identify and fill data gaps;
- fewer field mobilizations to complete cleanup;
- avoidance of stakeholder controversy and legal disputes due to inconsistent, ambiguous, and/or contradictory data sets (a consequence of high data variability stemming from uncontrolled soil heterogeneity); and
- reduced project lifetimes to achieve site completion, benefiting both site reuse and the workload for regulatory oversight.

These benefits far outweigh

- the additional efforts involved for proper field sample collection, sample processing and subsampling; plus
- the perceived additional effort to generate and evaluate sufficient QC data, which would normally be done by other parties as part of traditional laboratory analysis and data validation.

<u>Establishing when data is "good enough" to support confident decisions</u>
Soil contaminant data need to be accompanied by measures of its sampling and analytical variability to quality as "definitive data" (USEPA 2006). For this project, data obtained during field work will be evaluated for quality and sources of data variability in real-time. Knowing the respective contributions of sampling and analytical imprecision or bias allows proper targeting of corrective actions if an initial round of data is found to be not "good enough" to meet project goals for decision confidence and/or legal defensibility. Evaluating data quality in real-time and identifying and needed corrective actions early in field work avoids the danger of generating large amounts of data that are later found to be inadequate.

The simplest, and most objective, way to determine whether data are "good enough" is to incorporate all sources of data imprecision and known bias into a statistical confidence interval around the mean of a data set.

> The width of a statistical confidence interval is determined by an equation that takes into account the amount of data variability, how many data points are in the data set, and what level of statistical confidence is desired (such as 90 or 95%). The "upper confidence limit on the mean" (UCL) is the upper end of the confidence interval, and the "lower confidence limit on the mean" (LCL) is the lower end of the confidence interval. The higher the confidence level, the wider the interval.

Whether there is statistical confidence that a concentration result is truly below a screening level (or other type of decision threshold) is easily estimated by observing whether both the mean AND the UCL fall below the screening level (Figure 11-1).
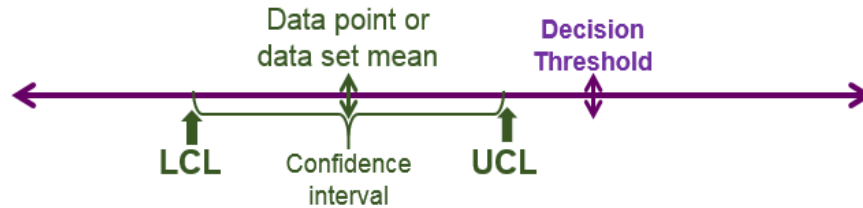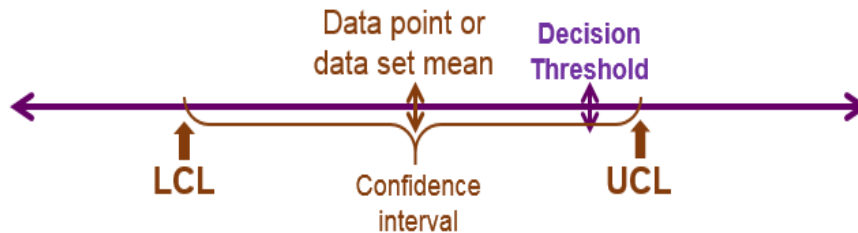

Figure 11-1. Statistical decision confidence: mean and UCL below decision threshold

Statistical confidence in the decision that a concentration result is <u>above</u> a screening level is based on observing whether both the mean AND the LCL fall <u>above</u> the screening level (Figure 11-2).
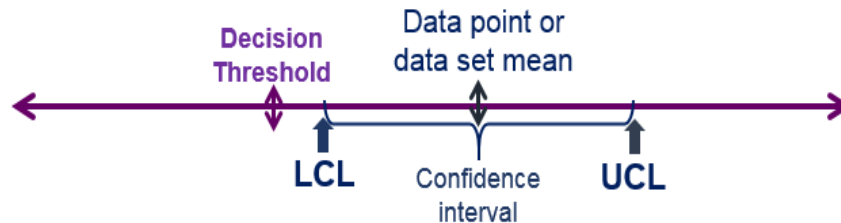
Figure 11-2. Statistical decision confidence: mean and LCL above decision threshold

When the mean and its UCL or LCL bracket the screening level, statistical uncertainty exists at the chosen level of decision confidence about whether the true concentration really is below or above the decision threshold (Figure 11-3).

Figure 11-3. Statistical decision uncertainty: confidence interval brackets decision threshold

The most important factor determining the distance between a mean and its upper and lower confidence limits is the degree of data variability. Higher levels of data variability widen the distance covered by a confidence interval. A wider interval increases the chance that it will bracket the decision threshold so that a statistically confident decision cannot be made. In other words, too much variability (i.e., poor precision or reproducibility) can cause the data to be "not good enough" to support a confident decision, even if the mean is well below the decision threshold. This is illustrated below with the results of a set of triplicate field samples from the same DU (Figure 11-4).

Triplicate Pb Results	Triplicate DU Samples Statistics			
	Mean	Std Dev	%RSD	95% UCL
36				
145	189	179	95%	492
387				

Figure 11-4. Field triplicate samples from a single DU used to calculate a 95% UCL for the DU. This example shows the effect that high data variability has on the distance between the mean and UCL.

The benefit of real-time data is the ability to discover a problem (such as an elevated UCL that inhibits confident decisions) while there is time to take immediate corrective action so that desired decision confidence can be obtained. Corrective actions, such as sample reanalysis or recollection with improved procedures, can improve data precision and narrow the confidence interval so that the entire confidence interval falls cleanly above or below the decision threshold.

Site-specific Practices to Support the Wilcox Project's Data Quality Strategy
The following describes general QC practices to establish adequate data quality in the context of the Wilcox project. More detailed aspects of the various QC checks are covered in Worksheet #12, in the Field Work Flow Attachments, and in the attached SOP Package).

- QC to ascertain the **precision of the <u>DU</u> sampling design**
  - Periodic independent triplicate incremental samples within the same DU. Optimally, field triplicate samples will agree within 20% RSD, but replicate precision is acceptable if it is sufficient for the 1-sided 95% UCL to be <200 ppm Pb. This QC is discussed in more detail in Attachment A, Section 3, Items 3 through 7, and in Worksheet #12 in the "Representativeness" section.
  - Corrective action for inadequate DU replicate performance would involve increasing the number of increments per DU incremental sample beyond 30.

- QC to ascertain the **reliability of <u>SU composites</u>** for establishing trends along transects
  - Initially, several sets of side-by-side paired SUs (SU couplets) will be collected, starting in the center and moving outward in the LSA.
    - Optimally, couplets results will be within 30% of the highest value, but the primary determinant of couplet data quality is that they provide consistency in the trend or boundary information provided.
    - Corrective actions for inconsistent couplets include enlarging the SU area and increasing the number of increments per SU.
      - The strategy for implementing these corrective actions to perfect the SU configuration is described in detail in Attachments A (LSA Field Work Flow) and B (EBA Field Work Flow)

- o   After optimization of the SU configuration, on-going SU QC will take the form of periodic side-by-side couplets. The location and timing of these couplets will be determined by
  - The need for accurate information when making critical decisions (such as at the 200-ppm boundary), and
  - To fill data gaps as indicated by geostatistical modeling.

- QC to ascertain that the **performance of the XRF instrument(s)** used in the project is(are) acceptable
  - o   Refer to Attachment C for operational performance measures for the TIIB Niton XL3t XRF (S/N 92959); and to Attachment D for the same for the ERT Olympus X-5000 XRF (S/N 202309). These measures include
    - Calibration status for Pb (indicates whether bias could be present in the XRF's Pb results);
    - Instrument limit of detection (LOD) and quantitation limit (QL) for Pb
    - Instrument precision
  - o   **Pre-field deployment QC** includes (see SOP Package, XRF Procedure 02.1.1)
    - Evaluation of the XRF's calibration status and linearity, i.e., determine any Pb-reporting bias in relation to a series of certified reference materials (CRMs) that evenly span the concentration range of interest
      - Ideally, the calibration curve for an XRF will have a slope of 1.0 ± 0.5 and a y-intercept near 0
      - If an XRF's calibration curve deviates significantly from these ideals, returning the instrument to the manufacturer for recalibration should be considered
    - Determination of instrumental precision for Pb for CRMs with concentrations relevant to project decision-making
    - Determination of instrument's Limit of Detection (LOD) for Pb in CRM materials
  - o   **On-going performance of the XRF** will be monitored using LCS control charts (see the SOP Package, XRF Procedure 02.1.2)
    - Three concentration levels (low, mid and high) will be chosen from the group of CRMs to use as laboratory control samples (LCSs).
      - The LCS set will be run on each XRF at least 4 times per day (at startup, before and after lunch, and at shutdown), with control charting of the results. Only Pb will be charted.
      - Examples of recent control chart performance for the two XRFs to be used in this project are presented in Attachments C and D.
    - At least twice a day a silica blank (99.995% purity) will be used to monitor the XRF for build-up of dust from samples or air deposition (but will be run more frequently if the unit is deployed outdoors in dusty conditions).

- QC to ensure **the quality of Wilcox sample data generated by XRF**
  - o   All XRF readings are recorded in a <u>logbook</u> specific to the XRF unit and to the project.
    - This logbook is used to correct any clerical errors (such as mistyped sample IDs) that appear in the raw XRF files or in the samples' RTeX forms.
    - Verification of XRF data after field deployment is complete will involve reconciliation between the 3 data records (logbook, raw XRF output, and sample RTeX forms)

- Completed logbooks will be scanned into pdf format for distribution to relevant parties and permanent storage as part of the Wilcox project's documentation.
- o Generating XRF Pb results on individual samples will involve taking <u>multiple readings</u> over the sample bag and entering each reading in a real-time Excel XRF (RTeX) form (a <u>statistical calculation</u> sheet, see SOP Package, XRF Procedure 01.3)
    - The within-sample statistical variability (measured as relative standard deviation, RSD) for a sample with particle size <2 mm (i.e., sieved through a 10-mesh sieve) is acceptable if
        - The 95% t-UCL/LCL below/above the screening threshold, and/or
        - The precision of multiple bag readings is <30% RSD
    - If the sample bag's data variability does not meet performance expectations, potential corrective actions include
        - Taking additional readings to enlarge the per-bag data set, and/or
        - Remixing the sample bag and reanalyzing it
    - The RTeX forms are retained as a permanent record of all XRF sample readings
- o Ensure that the <u>plastic bags used as XRF "read bags"</u> are free from significant interference for the target analyte
    - Use the Bag-Checker Tool to statistically compare CRM readings with and without 1 layer of plastic bag material (see SOP Package, XRF Procedure 02.1.3)
- o <u>XRF analyst proficiency</u> is monitored with duplicate sample analysis
    - A previously analyzed sample bag is reanalyzed as if it were a new sample
        - The duplicate analysis is entered into the designated block in the sample's RTeX form so that a t-test is automatically performed to determine whether the 2 sets of sample results are equivalent.
        - If the calculated p-value from the RTeX's t-test is <0.05, the 2 sets of readings do not agree.
    - If disagreement is found, the sample must be remixed and analyzed a third time to determine the correct bag concentration.
    - Data records will be corrected if the initial analysis was inaccurate.
- o Monitor for site- or sample-specific <u>matrix interferences</u> affecting the XRF readings
    - Instrument "noise" (i.e., pure analytical precision) can be measured through replicate readings taken in the same spot on the bag (i.e., repeated readings taken without moving the XRF window)
        - This QC check is performed periodically during the day on samples selected randomly, or if applicable, due to unusual behavior or appearance
        - The results are recorded in the sample's RTeX form
        - The results are used in calculations that partition sources of data variability (see the end of Worksheet #12)
    - The instrument-reported "noise" will also be monitored in the form of the sample-specific Limit of Detection (LOD), which is calculated automatically in the RTeX form
        - A sample LOD that is significantly higher than expected will trigger closer visual examination of the sample (color, particle characteristics) and possibly further testing of that sample
            - ✓ The sample may be remixed and reanalyzed by XRF

✓ The XRF spectrum for that sample may be compared to spectra from other site samples not showing elevated LODs to see if peak distortion can be observed
✓ The sample may be sent to a laboratory for further analysis, if warranted, to determine whether XRF-specific matrix interferences are present

- **Comparability analysis** to establish a quantitative statistical relationship between the XRF sample data and sample data reported by **a lab method for Pb** (such as ICP) is not anticipated for this project. The reasons for not doing comparability analysis:
    - o The usual relationship between XRF and ICP data is that there is reduced recovery of Pb from recalcitrant mineral matrices by ICP digestion procedures. This commonly causes XRF results (which measure total Pb) to be higher than corresponding ICP results (which measure solubilizable Pb).
    - o Because the Pb was released to the environment in a somewhat soluble form in both the LSA and EBA, it is expected that the XRF and ICP results would be numerically similar, since near 100% solubilization in the ICP's acid digestion could be possible.
    - o If not all soil Pb is solubilizable, using XRF results to establish the 200 ppm boundaries will add an element of conservatism to decisions (i.e., erring on the side of caution with regard to risk) as compared to using ICP results, and will reduce the costs and time required for the project.
    - o DU samples representing critical decisions will be archived so that ICP, bioavailability or mineralogical analyses can be performed if those tests are later determined to be advantageous.
- **Comparability evaluation** for the **two XRFs** to be used on the project (Figures 11-5 and 11-6) shows that they produce equivalent data sets, so that the Pb results from the XRF units can be used interchangeably.

The TIIB XRF instrument used for this work will be a Niton XL3t GOLDD Ultra. This is a handheld instrument [Figure 11-5(a)] that will be used in a stand to function in "bench-top" mode [Figure 11-5(b)], although occasionally it may be used in hand-held mode to shoot very large bags, or to "chase" contamination in the EBA.

Figure 11-5(a) Niton XL3t GOLDD Ultra; (b) mounted in stand

The second XRF is ERT's Olympus X-5000, a portable bench-top model (Figure 11-6). It is anticipated that one XRF will be dedicated to the LSA, and the other to the EBA in order to avoid confusion. However, since it is established that the performance

Figure 11-6. Olympus X-5000

of the Niton and the Olympus are nearly identical, the reported Pb results are entirely comparable no matter which instrument analyzes samples.

- The factors examined to draw the conclusion of direct instrument comparability for Pb are provided in Attachments C and D, and are summarized below:
  - Niton and Olympus calibration parameters are nearly the same:
    - Calibration regression slopes are 0.977 and 0.983, respectively
    - Y-intercepts are -3.1 and -4.6 ppm, respectively
    - R^2 are 0.9998 and 0.9996, respectively
  - Niton and Olympus LODs are both below 6 ppm (a longer XRF read time produces a lower LOD, the rule is that a quadrupling of the read time reduces the LOD by half):
    - Niton LOD = 5.5 ppm (30-sec read time)
    - Olympus LOD = 3.3 ppm (240-sec read time)
  - Niton and Olympus instrument/analytical precision are nearly the same despite the difference in the read time:
    - Precision at low Pb concentration (a CRM with 11 ppm Pb concentration)
      - Niton: 8.8% RSD (30-sec read time)
      - Olympus: 11.6% RSD (240-sec read time)
    - Precision at higher Pb concentration (a CRM with 300 ppm Pb concentration)
      - Niton: 1.1% RSD (30-sec read time)
      - Olympus: 1.5% RSD (240-sec read time)
  - The Niton and Olympus control charts for Pb have similar values for the mean, and the ±2 SD and ±3 SD lines (see for control chart graphics for Pb at 3 concentration levels for both instruments in Attachments C and D).

XRF data generated for the Wilcox Oil project will be considered "definitive" when the QC checks described above meet the limits provided in Worksheet #12, "Measurement Performance Criteria".

<u>General Soil Sampling Approach to Efficiently Achieve DQOs</u>
Sampling unit (SU) samples will be used to estimate approximate concentrations and trends so that more definitive **decision units (DUs)** can be placed to confirm the tentative 200-ppm boundaries indicated by the SU data. For the purpose of this QAPP, SUs and DUs are defined as follows:

**Wilcox Oil Sampling Units**
An SU in the Wilcox project is a very small area that is sampled with a 9 to 16 point composite.

- The SU sample result represents the <u>approximate</u> lead concentration at a "point," but the biasing effects of short-scale spatial soil heterogeneity are reduced by the compositing strategy (Figure 11-7).
- The Wilcox SUs are being used to gather spatial information on contaminant patterns and source areas.
  An SU data result is interpreted qualitatively or semi-quantitatively in relation to other SU data…is the concentration of one SU roughly higher, lower, or the same as other SUs around it?
- Linear arrangements of SUs (i.e., transects) will be used to search for the 200-ppm boundary (Figure 11-8).
- Statistical confidence in the SU's concentration estimate is not required.
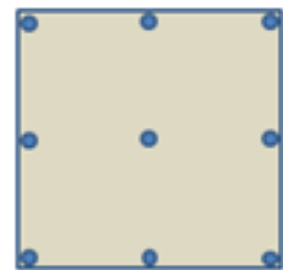
2 x 2 sq.ft. SU increment configuration

Figure 11-7.   SU example
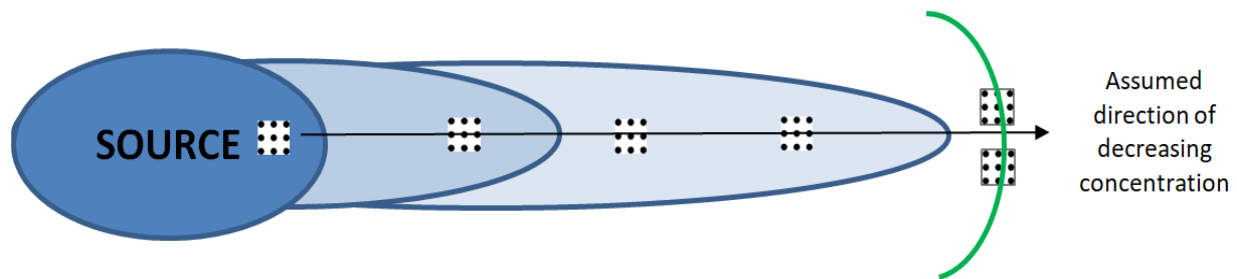
Figure 11-8. Example of an SU transect. Paired SUs (i.e., an SU couplet) will be used to confirm critical decisions, such as the tentative 200-ppm boundary (green arc).

SU couplets will be used as a field QC mechanism to establish and monitor the reliability of trend information provided by the SUs. If the initial SU configuration does not provide consistent information, different SU configurations will be tested at the start of field work until one is found. The SU configuration testing process is described in detail in Attachment A, Section 1, also see the graphical decision tree at the end of Attachment A.

Wilcox Oil Decision Units

A DU is a larger area that is sampled with a composite made of at least 30 increments in order to accurately represent the concentration of a unit volume of soil upon which a removal or exposure decision it to be made. The term for this design is "incremental sampling" or "Incremental Sampling Methodology" (ITRC 2012a).

- The larger area of a DU controls for between-sample heterogeneity at the spatial scale of decision-making. Knowledge of the spatial patterns of contamination within the DU unit volume is not needed.
- For the LSA, DUs are being used to definitively encircle the source area at the 200-ppm Pb boundary.
- For the EBA, DUs are being used to simultaneously 1) quantify Pb concentrations for exposure unit areas of compliant soil around the buildings, and 2) determine the boundaries of any Pb contamination exceeding 200 ppm. (Refer to Attachment B and its graphical decision tree).
  - DUs will be structured such that any small areas with elevated Pb due to piping leakage, spills, etc. will be excluded from DU areas. (Refer to Figure 11-9)
- Statistical confidence in the DUs' concentration results is required to demonstrate compliance with the 200-ppm action level and achieve risk assessment quality data.

Independent triplicate field samples are the field QC mechanism used for incremental sampling of DUs. This involves collecting 3 replicate (i.e., separate) samples of 30 increments each from the same DU (for a total of 90 increments in the DU), as illustrated in Figure 11-10. Triplicate field samples provide a QC measure of field sampling precision that determines whether the number of field increments is sufficient to control for the degree of within-DU field heterogeneity. This precision (i.e., variability) is also used to calculate the statistical upper confidence limit (UCL) that provides a basis for statistical confidence in the decision that a DU's concentration is below a numerical decision threshold.

Workload can be reduced by using a consensus variability term derived from a group of DUs and then the variability value to similar DUs. The consensus variability from replicated DUs is used to calculate a "predicted" UCL for those DUs having only a single incremental sample. In this way, statistical

confidence can be maintained for all DUs, but only a subset require the full effort of triplicate field samples. After the initial group of replicated DUs is done, occasional DUs are sampled with triplicate field samples as on-going QC and to monitor the reliability of the consensus variability value.
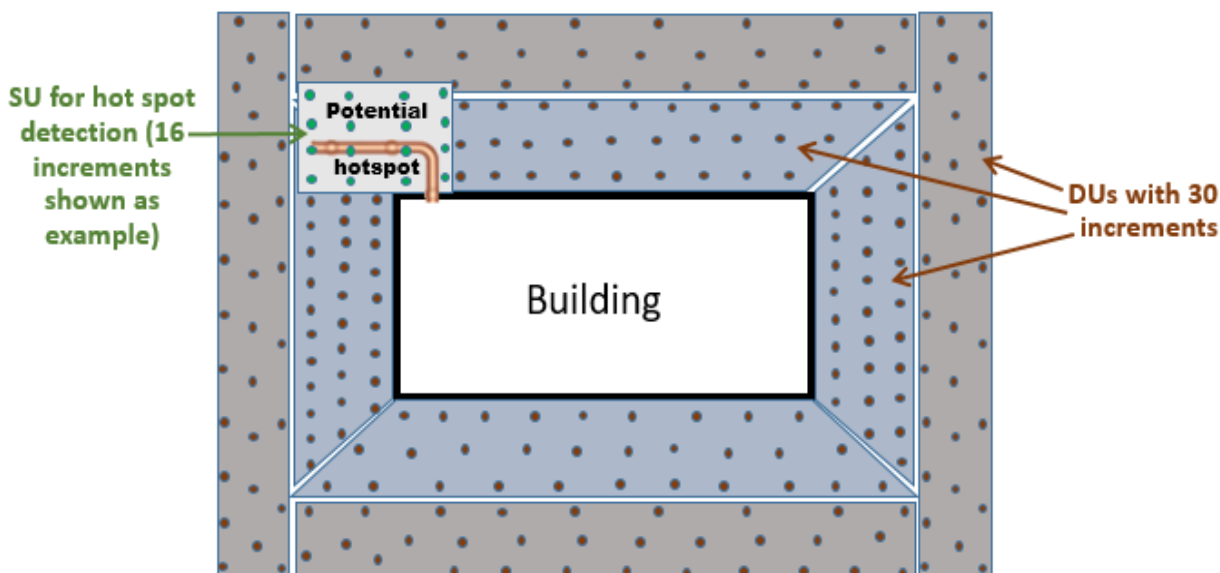

Figure 11-9. Example of DUs surrounding a building to confirm uncontaminated soil. An SU used to test a possible source area (piping) is illustrated in the upper left.
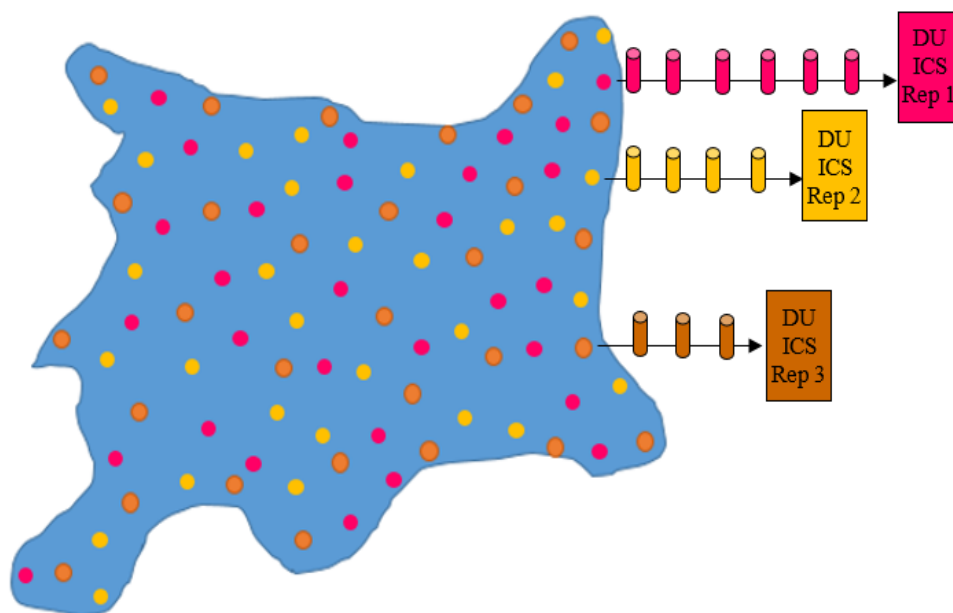

Figure 11-10. Illustration of triplicate field samples collected in a single DU.

Ecological risk assessment could be a use of these data. Different ecological receptors have different sized exposure units, but the Wilcox DUs are being structured primarily to serve compliance and mitigation goals. If individual DUs are too small to represent the exposure units for larger receptors, the

data from several smaller DUs can be combined to produce the mean and UCL for the larger combined area. This combining of DU areas is accomplished with a mathematical approach that is built into a programmed Excel spreadsheet (ITRC, 2017). Although obtaining the mean for the larger area involves simple area-weighting of the means of the individual DUs, simple weighting cannot be used to obtain the UCL for the combined mean. The UCL requires much more complex mathematics which can only be done using the specialized spreadsheet.

Decision Confidence Goals and Hot Spot Evaluation
It is desirable to keep false positive decision errors to a minimum to reduce the amount of compliant soil that is treated or removed from the site and requires disposal. It is desirable to keep false negative decision errors to a minimum to so that off-site Pb transport and receptor exposures are minimized.

- A false positive decision error for the LSA would be deciding that a boundary DU (i.e., the DU bridging the gap between the ends of two transects) exceeds the 200-ppm threshold, when in actuality it does not.
- A false negative decision error for the same would occur if a DU was considered to represent the outer edge of the >200 ppm volume, when in fact the DU's soil concentration exceeded 200 ppm and the outer boundary off the >200 ppm volume had not been identified for that location.
- A false positive decision error for the EBA would be deciding that a DU around the structures exceeds the 200-ppm threshold, when in actuality it does not.
- A false negative decision error would occur for the EBA if SUs missed a significant hot spot.

For the purposes of this project, a hot spot is any SU with a concentration >200 ppm. The process of evaluating an EBA hot spot to determine whether it is significant or not is discussed in Section 1, Item 1 of Attachment B, EBA Field Work Flow. A hot spot will be considered significant if any of the following are true:

- Any DU with a concentration >200 ppm;
- Any single small localized hot spot on one side of a structure having an area larger than 4 sq.ft. in the 0-6" interval and a concentration greater than 200 ppm;
- A hot spot of any size that has a Pb concentration higher than 400 ppm;
- Two or more distinct hot spots of any size on one side of a structure; and
- A hot spot >200 ppm of any size in the 6-24" depth interval.
  - o A hot spot smaller than 4 sq.ft. in the subsurface may be considered significant if the circumstances suggest the detection may represent a subsurface migration pathway away from a ethyl-Pb spill or leak that pooled in the subsurface.
  - o Such a detection may require additional subsurface sampling to determine whether it represents a migration conduit transporting Pb off-site.

Insignificant hot spots (i.e., an area shown to be less than or equal to 4 sq.ft. in the 0-6" interval with a concentration between 200 and 400 ppm) will not be flagged or investigated further. Significant hot spots will be flagged and mapped so that their disposition can be considered during remedial design.

Error Rate Goals: With the many QC checks built into both the DU/SU sampling designs and the XRF analytical protocols, the false positive and false negative decision error rates for the Wilcox project are expected to be close to zero.

- DUs:
  - o The false negative decision error rate for DUs will be held to 5% or less by using the actual (or predicted) 95% UCL based on triplicate field samples within a single DU;

- o The false positive rate is difficult to quantify, but is minimized by using samples that represent the entire area of a decision unit, and by using sufficient increments as documented by the QC measured by the triplicate DU samples.
- SUs:
  - o The false negative rate for individual SU samples will be held to 5% or less by using the actual 95% UCL for an SU sample bag, calculated by the RTeX spreadsheet from the replicate readings on each sample bag;
  - o Both the false positive and false negative decision error rates for the representativeness of SUs will be controlled by
    - By optimizing the SU configuration and periodic QC (using SU couplet sets) that ensure that geostatistical modeling of SU results is not biased by non-representative SU results.
    - Quantifying the uncertainty in geostatistical modeling: where necessary to maintain 80% confidence in the modeled 200-ppm boundary, additional SUs will be placed at locations to fill data gaps leading to modeling uncertainty.

References for Worksheets 10 and 11

Interstate Technology Regulatory Council (ITRC) (2012a) February. Technical Guidance Web Document: Incremental Sampling Methodology: Representative Data, Confident Decisions. http://www.itrcweb.org/ISM-1/

ITRC (2017) Incremental Sampling Methodology. ISM-1. Washington, D.C.: Interstate Technology & Regulatory Council, Incremental Sampling Methodology Team. Calculator for the Weighted 95% UCL for a Combined DU from Smaller Several DUs. Original date 2012, updated in 2017.

Lachman, Arthur (1931) Chemistry of the Doctor Sweetening Process. *Industrial and Engineering Chemistry Vol. 23*, No. 4, pp. 354 – 357. URL: http://pubs.acs.org/doi/pdf/10.1021/ie50256a004

Kady, Tom (2015) Figure from 15Dec2015 conference call presentation materials provided by the USEPA Environmental Response Team to the Wilcox Oil Site project team.

USEPA (1999) *Correct Sampling Using the Theories of Pierre Gy*. Environmental Sciences Division, Office of Research and Development. Las Vegas. Technology Support Project. March. URL: http://www.itrcweb.org/ism-1/references/csutpg.pdf

USEPA (2006) Applicability of Superfund Data Categories to the Removal Program. Quality Assurance Technical Bulletin No. 2 of 3. Office of Solid Waste and Emergency Response. OSWER 9360.4-21FS. July. URL: https://www.epa.gov/sites/production/files/2015-10/documents/dc-qatib_final_for_sig2.pdf

USEPA (2016) Recommendations for Sieving Soil and Dust Samples at Lead Sites for Assessment of Incidental Ingestion, OLEM Directive 9200.1-128. July. URL: https://semspub.epa.gov/work/HQ/100000133.pdf

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Soil
Analytical Group or Method: XRF (Lead)
Matrix Concentration Level: Pb: 10 – 5600 ppm and above, possibly to percent levels

Data Quality Indicator (DQI)	QC sample or measurement performance activity	Measurement Performance Criteria
XRF Analytical Precision (instrument only) Evaluates instrument stability and repeatability over time under the ambient operating conditions	Replicate instrument measurements: Sample bag not moved between replicate measurements. Precision calculated using the dedicated "Instrument Duplicates Calculator," or the data can be entered into a sample's RTeX form (in data blocks marked for this purpose).	• If the Instrument Duplicates Calculator is used, acceptance is that the 2nd replicate reading be within the 95% confidence interval (CI) of the first reading as described in *Procedure 2.1.1 Assessing Instrument Precision and Bias* will be used for data recording and interpretation. • If the RTeX form is used, there is no built-in calculation of acceptance criteria as in the Instrument Duplicates Calculator, but the %RSD for instrument replicate readings should be significantly less than the %RSD calculated from readings taken over the bag.
Sample Analysis Precision Evaluates analyst performance for proper mixing of sample bags prior to analysis.	A previously analyzed sample bag is reanalyzed anew. This may be done by the same person or by another person who did the first analysis.	• Acceptance criteria are built into the RTeX form with a pre-programmed t-test for a difference between means. The t-test function automatically reports the t-test p-value and whether the duplicate analyses agree at 95% confidence. • If the test fails, both bags should be remixed and reanalyzed. • For this project, the average of the final readings is considered to be the bag sample's concentration.
XRF Instrument Analytical Bias and Linearity	Pre-project: Regression analysis of 6 or more CRMs establish XRF calibration curve to 1400 ppm for Pb.	No systematic bias for XRF results vs. CRM certified Pb value (verifiable by a Wilcoxon Signed Rank paired statistical test). If bias is present, a mathematical correction of the bias might be possible. Demonstrate linearity of calibration curve to 5600 ppm Pb (highest CRM available).
XRF Measurement Precision and Instrument Stability	LCSs (selected Certified Reference Materials in XRF cups with certified values for Pb), analyzed several times/ day & plotted on paper control charts.	A set of 3 Pb LCSs (with low, mid & high concentration levels selected from the CRM set) will be used. Results within control chart limits (±2 standard deviation).

Cross-contamination check	99.995% Silica blank in XRF cup	Pure silica blank should be ND for Pb
Analytical Interferences	Interference (positive or negative bias) due to plastic bag material	No statistical difference indicating <u>significant</u> interference by sample reading bag plastic for any of the analytes of interest (Pb). The significant of interference is assessed using the Bag Checker spreadsheet tool (see *Procedure 2.1.3 Assessing Interference from Plastic Sample Bags*)
Analytical Sensitivity	XRF sample-specific LOD = 3 x the instrument reported standard deviation on each sample reading.	Instrument limits of detections (LODs) for Pb should be at least 20 ppm on CRM matrices. Calibration curve slope should be 1.0 +/- 0.1 for Pb CRMs.
Completeness	Real-time decision-making detects when data are missing or data gaps exist	100% (no missing or lost data). Missing or aberrant sample data will prompt sample reanalysis or recollection.
Field Sample Representativeness	Sample representativeness will be assessed for • SUs: by using side-by-side couplets. After testing and optimization of the SU configuration, periodic SU couplets will be used to double-check ambiguous findings or to resolve uncertainty in geostatistical modeling. • Boundary confirmation DUs: a frequency of at least 20% triplicates overall. Replication will be targeted to control decision uncertainty. The first 2 boundary DUs sampled will automatically get triplicates, and the variability information will be used to establish the Criterion Value described in Attachment A, Section 3, Item 3.	• SU couplet results will optimally be within 30% of the highest value, but the primary quality determinant will be whether the couplets provide consistent information about Pb concentration trend or the 200 ppm boundary. If couplets frequently give contradictory information, further revision of the SU configuration will be considered. • The appropriate SU configuration will be determined at the start of field work in the LSA with one of more rounds of testing. Four sets of 0-6" depth side-by-side SU couplets will be placed to span a high to low concentration range along a transect. All 8 samples will be analyzed and evaluated for information reliability. If any information given by these samples is inconsistent (e.g., one member of the couplet indicates increasing concentration and the other indicates deceasing concentration), the SU configuration will be revised and retested as described in Attachment A, Section 1. • The first 2 to 3 SUs to be collected after SU optimization will be also be couplets to ensure SU reliability, as described in Attachment A, Section 2, Item 1. • If a satisfactory SU configuration cannot be found, potentially all SU samples will need to be collected as couplets to control decision error. • DU field triplicates will optimally agree within 20% RSD. However, if

		they do not, replicate sample precision is still acceptable if it is sufficient for the 95% t-UCL to be < 200 ppm. • The Criterion Value for each single DU result must be <200 ppm
Analytical Sample Representativeness	Particle size will represent the exposure pathway	The exposure pathway evaluation will use data from the <100-mesh (150 micron) particle size per the latest Superfund guidance (EPA, 2016). Goals for analytical precision for Pb over a sample bag is <15%RSD for particle size fractions <60-mesh, and <30%RSD for <10-mesh samples.
Sample Result Uncertainty	Repeated XRF readings over the sample bag controls subsampling error to achieve 95% statistical confidence for the UCL or LCL (as applicable). A programmed Excel worksheet ("the RTeX form") provides the statistical calculation in real-time.	The reported bag result will be accompanied by the 95% statistical confidence interval. The bag conc used may either be the mean or the 95% UCL/LCL, depending on how the data result is to be used. On periodic field replicate samples, the sources of data variability will be partitioned into instrumental analysis, subsampling (within-sample heterogeneity), and field heterogeneity. See RTeX illustration and field heterogeneity equation below this table.
Project Decision Confidence False positive and false negative decision error rates	For DUs: Actual or predicted* 95% UCLs based on DU triplicate incremental samples For SUs: • Actual 95% UCL on the sample bag mean (calculated from replicate XRF readings over the bag) • SU couplet QC shows SUs are providing reliable information • Geostatistical modeling uncertainty quantitation *See Attachment 1, Section 3, Item 3.	DUs: Maximum of 5% statistical likelihood of decision error achieved by decision-making that requires the 95% DU-UCL must be <200 ppm Pb, or step-out to a new DU. SUs: An SU sample with a UCL >200 ppm is considered to exceed 200 ppm even if the bag mean is <200 ppm. The process of optimizing the SU configuration is described in Attachment A, Section 1. SU couplet QC is described in Attachment A, Section 2, Items 1 and 4. Achievement of >80% confidence in the geostatistical modeling of the 200-ppm iso-concentration line is accomplished by placing additional SUs or SU couplets to fill data gaps in locations indicated by the model.

Illustration of RTeX form (real-time statistical calculator & QC tool for XRF)

Project: Colorado Smelter, Community sampling; DMA

Property ID: FY1504

This sheet records XRF concentration results for DU bag

<60-mesh particle fraction read

Operator: D. Crumbling

Read Bag lot = PO262010

Date: 18-May-2015

As mean	As UCL	As %RSD	Pb me	Pb UCL	Pb %RSD
14.0	17.3	24.8	45.0	49.1	9.7

DU or Bag ID: $1504-FY-1218-01   Element: As

Replicate Bag Readings	Time	Reading No.	Run Time (sec)	Instrumt Result (ppm)	Instrumt Error (as 1 Std Dev)	Note?
Sample or Location ID =						
Replicate reading 1	1323	239	30	11.57	2.35	
2	1328	240	30	11.96	2.31	
3	1331	241	30	15.10	2.34	
4	1336	242	30	19.61	2.36	
5 (optional)	1340	243	30	11.68	2.46	
6 (optional)						
7 (optional)						
8 (optional)						
9 (optional)						
10 (optional)						
			Mean	14.0		
			SD	3.47	**Ttl %RSD**	24.8
			n =	5		
		ProUCL distribution =				
	2-sided	Smple 95% t-LCL =	9.7			
	2-sided	Smple 95% t-UCL =	18.3			
	1-sided	Smple 95% t-LCL =	10.7			
	1-sided	Smple 95% t-UCL =	17.3			
	1-sided Smple 95% Chebyshev LCL =		7.2			
	1-sided Smple 95% Chebyshev UCL =		20.7			
subsampling error =		18.15		instrument error =	16.91	as %RSD

DU or Bag ID: $1504-FY-1218-01   Element: Pb

Replicate Bag Readings	Time	Reading No.	Run Time (sec)	Instrumt Result (ppm)	Instrumt Error (as 1 Std Dev)	Note?
Sample or Location ID =						
Replicate reading 1	1323	239	30	45.4	2.96	
2	1328	240	30	44.5	2.89	
3	1331	241	30	46.3	2.89	
4	1336	242	30	38.3	2.82	
5 (optional)	1340	243	30	50.4	3.11	
6 (optional)						
7 (optional)						
8 (optional)						
9 (optional)						
10 (optional)						
			Mean	45.0		
			SD	4.35	**Ttl %RSD**	9.68
			n =	5		
		ProUCL distribution =				
	2-sided	Bag 95% t-LCL =	39.6			
	2-sided	Bag 95% t-UCL =	50.4			
	1-sided	Bag 95% t-LCL =	40.8			
	1-sided	Bag 95% t-UCL =	49.1			
	1-sided Bag 95% Chebyshev LCL =		36.5			
	1-sided Bag 95% Chebyshev UCL =		53.5			
subsampling error =		7.15		instrument error =	6.53	as %RSD

Progression of mathematical relationships to partition sources of variability to soil data

Total variability = Field variability + Subsampling variability + Analytical variability

Total variance = Field variance + Subsampling variance + Analytical variance

$$SD_{Total}^2 = SD_{LCS-instrument}^2 + SD_{analytical\ subsample}^2 + SD_{betw-IS\ samples}^2$$

$$(Total\ \%RSD)^2 = (Field\ \%RSD)^2 + (Subsampling\ \%RSD)^2 + (Analytical\ \%RSD)^2$$

$$Field\ \%RSD = sqrt((Total\ \%RSD)^2 - [(Subsampling\ \%RSD)^2 + (Analytical\ \%RSD)^2])$$

Example of an Excel calculator that partitions variability from QC data

Park #1		
STATION_LOCATION	SAMPLE_ID	BaP
Park #1 - LabRep1/FieldRep1	15412000	210
Park #1 - LabRep2	15412008	200
Park #1 - LabRep3	15412009	210
Park #1 - FieldRep2	15412001	200
Park #1 - FieldRep3	15412002	200

BaP for Park #1				
Total Variability (Use Lab Rep1 only)				Relative Variance
Mean		SD	RSD (%)	
203.3		5.8	2.8	8.1
Processing/Subsampling + Analytical Variability				
Mean		SD	RSD (%)	
206.7		5.8	2.8	7.8
Analytical ONLY (from LCSs)				
Mean		SD	%RSD	
101.0		2.0	0.02	0.0004
Sample Processing & Subsampling Variability ONLY, as %RSD				
sqrt(Proc rel.var. - Anal rel.var.)			2.8	
Field Sampling Variability ONLY, as %RSD				
sqrt(Ttl rel.var-(Process+Anal rel.var)			0.51	
Summary	Component SDs		Component RSDs	
Field	1.0	**Field only**	0.51	%
Processing	5.8	**Processing only**	2.8	%
Analytical	2.0	**Analytical only**	0.02	%
		Total RSD	2.8	%

QAPP Worksheet #13: Secondary Data Uses and Limitations
(UFP-QAPP Manual Section 2.7)
(EPA 2106-G-05 Chapter 3: QAPP Elements For Evaluating Existing Data)

Data type	Source	Data uses relative to current project	Factors affecting the reliability of data and limitations on data use
Concentrations of metals in soil and the kriged (contoured) iso-concentration maps resulting from that data	ERT Investigation: Lockheed Martin SERAS contract (LMS). 2016. Trip Report: November 30 through December 16, 2015, Wilcox Oil Company Superfund Site. Draft. Work Assignment No. SERAS-277. 15 April Raw XRF Data available in Scribe database Maps and figures in report are based on kriged and contoured data.	The 2015 kriged data for the EBA establishes a preliminary area of interest for further delineation in this study. The 2015 kriged data for the LSA are being used to establish a point of comparison for positioning transects and determining step out distances for SU samples along transects.	*In situ* readings by handheld XRF may not account for small scale heterogeneity of contaminants, and grain size segregation of contaminants. The resulting data variability can produce misleading contour lines. Many samples had extremely high concentrations (% level) and were out of the effective calibration range of the instrument. Kriged and contoured data is an interpolation of concentrations between sample points and does not necessarily represent actual concentrations. There were few low concentration samples to define the 200 ppm boundary.
Screening sample locations and site features	SERAS Investigation Report (referenced above)	Selection of sample locations to establish Pb concentrations in soils.	GPS accuracy and precision; registration of air photos and changes in site features. Identifying and re-occupying previous sample locations and the 200 ppm contour intervals will be approximate.

QAPP Worksheet #14/16: Project Tasks & Schedule
(UFP-QAPP Manual Section 2.8.2)
(EPA 2106-G-05 Section 2.2.4)

Activity	Responsible party	Planned start date	Planned completion date	Deliverable(s)	Deliverable due date
XRF Instrument Evaluation Period	EPA TIIB	N/A	Was completed as part of prior XRF field projects	Performance summary	Summary provided in Worksheet #11 of this QAPP
XRF QC control charts and RTeXs	EPA TIIB	August 4, 2017	August 11, 2017	Updated Control Charts for Pb if needed; RTeX template adapted to Wilcox project needs	Sampling Start date **October 23, 2017**
Mobilization	EA TIIB, ICF, ERT	October 16, 2017 or earlier	One week prior to field event start date October 23, 2017	Equipment Checklists, Supplies (sent to hotel/site)	Upon completion
Sample collection-soils	ICF, ERT,	Sampling Start date October 23, 2017	est. November 11, 2017 (18 field days)	Field notes	1 week after demobilization
Sample analysis-soils	Sample Prep and Analysis Lead, Technical Lead	est. October 24, 2017	est. November 10, 2017 (18 days)	Raw RTeX spreadsheets, scanned log books, raw XRF download files	1 week after demobilization; post to shared FTP site
Geostatistical modeling of SU data	ICF	est. several days into field work starts	final day of field mobilization	Real-time electronic kriged maps of SU data to guide further sampling of the LSA and EBA	Final maps due 4 weeks after demobilization for inclusion in summary report.
Verification	EPA TIIB	1 week after demobilization	2 weeks after demobilization est. October 20 2017	Reconciled RTeX sheets; clerical corrections in XRF downloads;	3 weeks after demobilization (December 4, 2017)
Usability assessment	Project Team	October 2017	November 2017	Summary of XRF & sampling QC performance	Summary included in Draft Report
Summarize data	TIIB with support from ICF	TBD	TBD	Draft Report	December 2017 (est)

QAPP Worksheet #15: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Soil
Analytical Method: Niton XRF
Concentration level (if applicable): variable from 10 ppm to over 100,000 ppm

Analyte	CAS Number	Project Action Limit (i.e. Decision Criteria) (ppm)	Project-Required Quantitation Limit (ppm) (1/5 of decision threshold)	Achievable XRF Limits	
				Instrument LOD (ppm)	Quant Limit (defined as the lowest conc where instrument precision is +/- 10%
Lead	7439-92-1	200	40	5.5 (with 30-sec read time)	~10 ppm (with 30-sec read time)

Matrix: Soil
Analytical Method: Olympus XRF
Concentration level (if applicable): variable from 10 ppm to over 100,000 ppm

Analyte	CAS Number	Project Action Limit (i.e. Decision Criteria) (ppm)	Project-Required Quantitation Limit (ppm) (1/5 of decision threshold)	Achievable XRF Limits	
				Instrument LOD (ppm)	Quant Limit (defined as the lowest conc where instrument precision is +/- 10%
Lead	7439-92-1	200	40	3.3 (with 240-sec read time)	14 (with 240-sec read time)

QAPP Worksheet #17: Sampling Design and Rationale
(UFP-QAPP Manual Section 3.1.1)
(EPA 2106-G-05 Section 2.3.1)

The release mechanism and characteristics of the contaminants are different for the EBA and LSA so different approaches will be used. Two exposure zones (0 to 6 inches for human health and 0 to 24 inch for ecological) will be addressed in each sampling rationale.

This sampling design employs two types of field composites: composites collected to represent sampling units (SUs), and incremental samples that represent decision units (DUs). Generically, an SU is any volume of soil represented by a sample. By definition, a DU is also an SU. However, a DU is a special type of SU (see text box below). So, although all DUs are SUs, not all SUs are DUs. In this QAPP, SUs are defined for the narrow purpose of gathering spatial information about concentration patterns and trends. DUs will provide confirmation of the patterns suggested by SUs. Since SUs and DUs have different purposes, they will have different numbers of increments and encompass different soil volumes (see text boxes).

> In this QAPP, a **Sampling Unit** is a mass or volume of soil for which the goal is to obtain information about the spatial distribution of contaminants (primarily trend information). These SUs will employ smaller volumes and fewer increments than the DUs because the SU soil concentration need only be approximated. SUs used in this project will be comprised of 9 to 16 increments collected from a 4-to16-square foot area for a specific depth interval.

All samples will be collected using a device (such as a corer) that includes the 0-6" depth interval. Not all sample collections will include the 6-24" subsurface interval. The decision to collect the subsurface interval will be made on-site based on the levels of Pb found in that interval, and the degree of uncertainty in drawing the boundary line and determining exposure point concentrations.

Since these are incremental samples, the bottom 18 inches (representing the 6-24" depth interval) and the top 6 inches (representing the 0-6" depth interval) of all cores from a single DU or SU will be gathered into two separate plastic storage bags. One storage bag will contain all the increments for the 0-6" depth interval, and the other will contain all the increments for the 6-24" interval.

Worksheet #17 supplies a general overview of the sampling strategy to be deployed for the lead-contaminated areas found by the 2015 ERT field efforts, namely the Lead Sweetening Area (LSA) and the Ethyl Blending Area (EBA). The detailed designs for sampling each area is covered in Attachment A (LSA Field Work Flow with decision tree graphic) and Attachment B (EBA Field Work Flow with decision tree graphic).

> A **Decision Unit** is the smallest volume of soil for which a distinct risk, cleanup or compliance decision will be. Since an accurate estimate of DU concentration is needed, DUs are sampled using a default of 30 increments. In addition, independent replicate field samples are used to provide QC and statistical confidence in the DU concentration estimate. In this study, DUs are used to confirm the 200-ppm boundary suggested by SU data, and to provide data suitable for risk assessment. A DU will cover a specific depth interval for an area ranging from 1000 to 6000 square feet. If larger DUs are needed for exposure assessment, results from smaller DUs will be combined mathematically to cover a larger area.

Lead Sweetening Area (LSA)

The LSA in known to contain an extremely high lead (Pb) concentration in its center (referred to as the "source") with concentrations decreasing with distance from the source. The general approach will

be to determine the lateral extent of Pb concentrations >200 ppm, and whether/ where Pb has migrated vertically to the deeper soil interval (the 6 to 24-inch zone).

SU samples that represent the surface and subsurface depth intervals will be collected to determine the spatial pattern of Pb contamination. However, not all subsurface SU samples may be analyzed in those locations where Pb contamination is shown to not have migrated downward. Those samples will be archived for analysis at a later date if needed. If/where there has been downward migration, the cleanup footprint for the 6 – 24" zone will be delineated using 6-24" SU sample data.

Surface soil delineation. Prior to sampling, the approximate location of the 200 ppm boundary modeled by the 2015 ERT work will be marked in the field using GPS and flagging. Sampling will start by testing the default SU configuration (2x2 sq.ft. area and 3x3 increment arrangement) on a transect of couplet SUs that begins in the high concentration source area. These side-by-side couplet SUs will be compared for consistency (Figure 17-1).
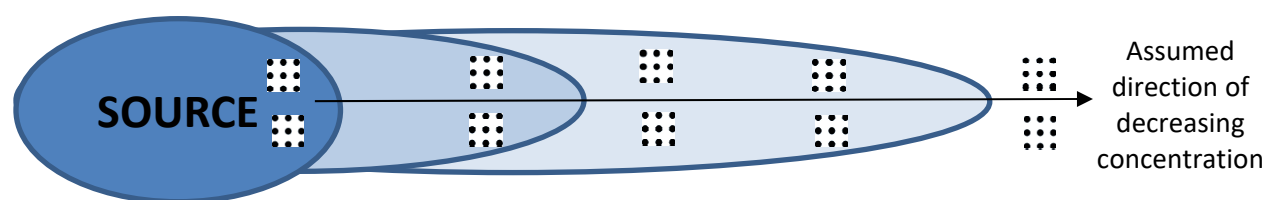

Figure 17-1. Transect to test the adequacy of SU configuration using "Test-SU Couplets"

If the performance of the default SU configuration is inadequate, the configuration will be optimized as described in Section 1 of Attachment A ("LSA Field Work Flow"). The horizontal 200 ppm boundary for surface soils (0 to 6 inches) will be delineated using the optimized SU configuration along radial transects (Figure 17-2). Except for periodic field QC checks, singlet DUs will be used along the delineation transects.

Attachment A (LSA Field Work Flow), Section 2 describes how early SU samples will be used to assess the reliability of the 2015 modeling for predicting the location of the 200-ppm boundary (the pink line in Figure 17-2).

If the 2015 modeling is found to be reliable, transects will be sampled first at the modeled 200-ppm boundary. New SUs will be placed along the transect inward or outward based on the first SU results. When an SU is believed to have found a 200-ppm border, a second SU will be placed 5 to 10 feet from the first to form an SU couplet as a QC check.
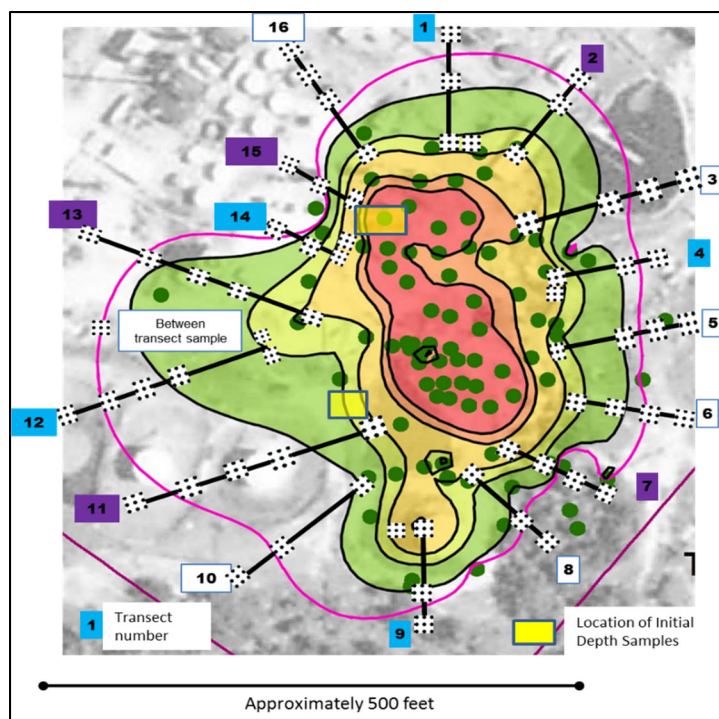

Figure 17-2. Radial transects of SUs in the LSA

If early SUs find that the 2015 modeled concentrations are not reliable, transects will be sampled from the source outward until the 200-ppm boundary is located. Early data will also be used to decide the degree of subsurface sampling needed to provide confidence in removal and risk decisions.

The anticipated maximum number of delineation transects (n = 16) are depicted in Figure 17-2. Real-time geostatistical modeling of SU data (using a contouring/kriging package such as EVS©) will guide actual placement and the number of transects needed to produce confident modeling of the 200-ppm iso-concentration line.

Once transect SUs bracket the tentative 200-ppm boundary on each transect, additional SUs may be placed between transects as needed to improve spatial coverage between transects to reduce modeling uncertainty. The location of the developing 200-ppm line will be flagged in the field.

DUs will then be placed to cover the space between transects, with the inner DU edge approximating the 200-ppm boundary modeled from the SU data (Figure 17-3). DUs will be approximately 10 feet wide.
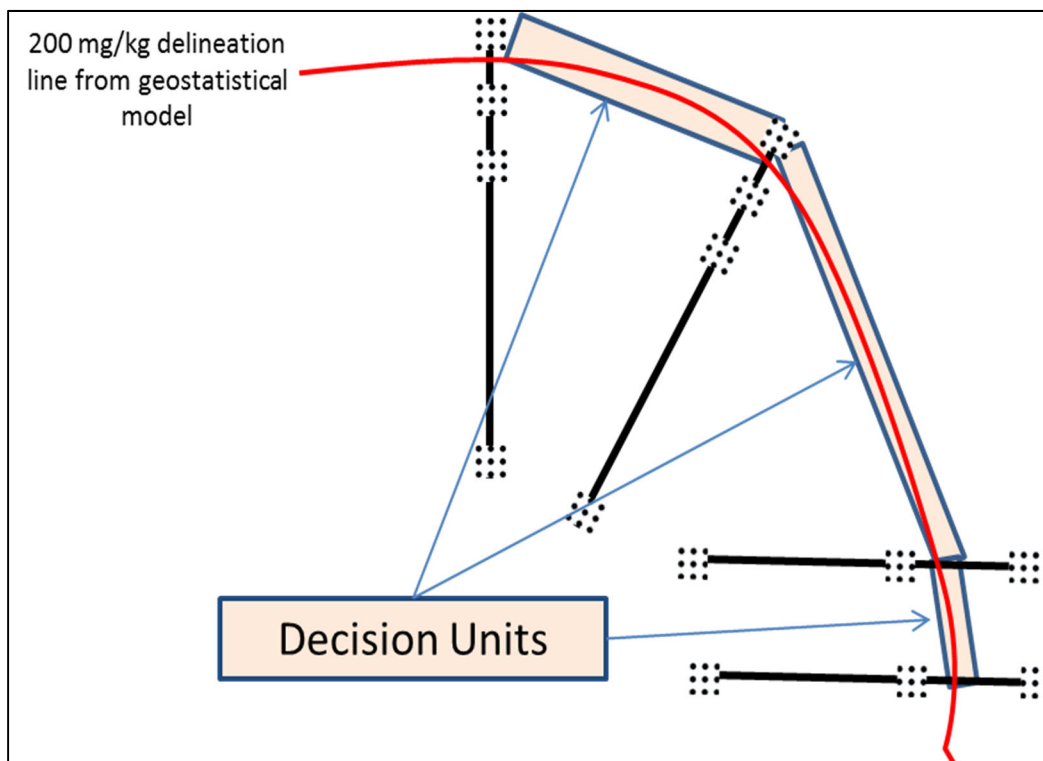
Figure 17-3. Example Decision Units to confirm the modeled 200-ppm boundary

The DU boundary will be considered confirmed when the actual or predicted 95% UCL on the DU mean is <200 ppm. A mathematical mechanism was developed to predict the 95% UCL based on a single DU-IS sample result. This mechanism will reduce the sample collection and processing workload, while ensuring sufficient decision confidence. This mathematical decision strategy to predict a DU's UCL is described in Attachment A, Section 3, Item 3.

Periodic triplicate DU-IS samples will be used as QC and for any DUs for which the conclusion of the predicted UCL is in doubt, as described in Attachment A, Section 3, Item 5.

If a DU fails the 95% UCL, the DU footprint will be moved outward and the new DU sampled, as illustrated in Figure 17-4.
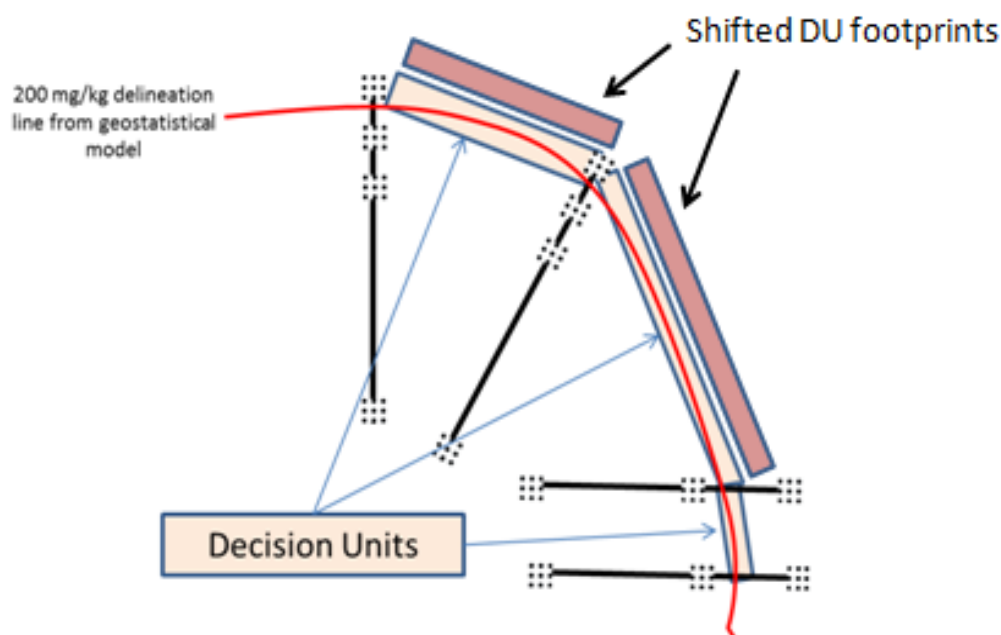

Figure 17-4. New DUs are placed outward if an initial DU's UCL is >200 ppm

Quality control checks will monitor both SU and DU precision during the investigation. These activities and the QC acceptance criteria are discussed in detail in Worksheet #12 ("Measurement Performance Criteria"), in the section for "Field Sample Representativeness."

Ethyl Blending Area

The sampling design reflects the limited knowledge and data regarding the history, source and existing conditions at this area. This approach assumes that the two buildings and their immediate proximity is the most likely source area, and concentrations decrease away from the source. However, we recognize that the ERT contour map (Figure 17-5) is based
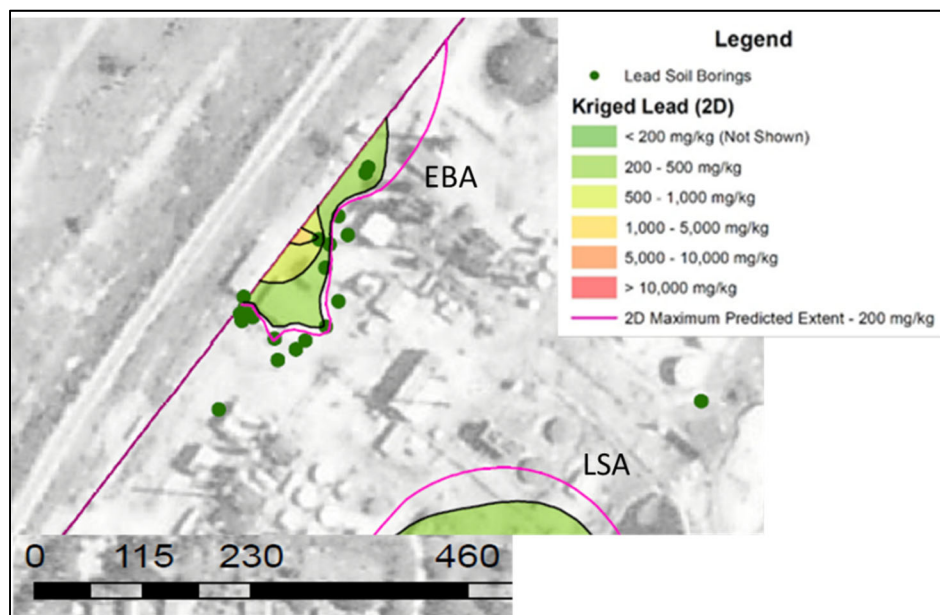

Figure 17-5. ERT 2015 contour map of Pb concentrations in the EBA

on a limited number of samples, only one of which suggests a high concentration away from the rear of the building (to the northwest).

Determination of Potential Source Areas in the EBA. The sampling strategy for determining if any significant areas are present that have lead concentrations greater than 200 ppm will begin with a visual evaluation of existing conditions for indications of potential release mechanisms. Potential source areas (PSAs) could include material storage areas, product transfer points (such as piping or valves), release areas adjacent to doors, and any stained or distressed areas near the building. The hand-held XRF may be used in *in situ* mode to test the soil surface for contamination in these areas as a means to

- test and evolve elements of the EBA's conceptual site model,
- assess the potential EBA sampling workload, and
- refine the EBA sampling strategy.

However, all PSA locations testing negative by *in situ* XRF must still be sampled with sampling units (SUs) encompassing the two depth intervals. If the lead concentration of any SU sample in a PSA location exceeds 200 ppm (as estimated by the 95% t-UCL on the SU sample's bag mean), additional SUs will be collected around it to ascertain the extent and patterns of concentrations greater than 200 ppm in that vicinity.

An area of soil with Pb concentrations >200 ppm will be considered significant if

- the volume of soil exceeding the 200-ppm Pb threshold is larger than 4 sq.ft. in either the 0-6" or 6-24" depth interval, and it has a Pb concentration greater than 400 ppm, or
- there is more than 1 SU along the side of a structure that has a concentration >200 ppm, or
- the RPM judges it significant using professional judgment.

Attachment B (EBA Field Work Flow) contains details for how the EBA investigation should progress. In general, if SU samples find that large areas of contamination >200 ppm exists in both depth intervals, 30-increment DUs (at both depth intervals) will be placed just outside the SU-identified area to confirm the 200 ppm cleanup boundary. If contamination only occurs in the surface interval, 30-point DU samples will only encompass that interval. If all SU samples show Pb levels <200 ppm, the entire side of the building will be bordered by a 10-ft wide, 30-increment DU (3 rows of 10) sampled at the 2 depth intervals and with triplicate DU-ISs. No matter what the outcome, the project manager will be supplied with exposure point concentrations for the 0-6" interval and the 0-24" interval. If applicable, a cleanup footprint and estimated cleanup volume will be supplied.

Sampling inside the east building. During field reconnaissance the field team observed that the eastern building appeared to have a dirt floor. At least one DU will be developed inside the building to sample each depth interval (0 to 6 inch and 6 to 24 inch).

Evaluation of North-South trend. The SERAS investigation data were used to develop a concentration contour map that shows an elevated area of contamination. This area will be investigated with a string of exploratory (0-6 inch depth) SUs to determine whether the elevated in situ shots recorded by the SERAS team represent areas of significant concentration. If areas of significant concentration are found, SU samples at both depth intervals of interest will be collected. If necessary, a contaminated area will be bounded by DUs to establish a 200-ppm perimeter. Step-out DUs will be used if necessary.

All samples will be processed per XRF Procedure 1.2 (see SOP Package). SU samples are sieved to 10-mesh prior to XRF analysis. DU samples may initially be sieved and analyzed at 10-mesh for exploratory purposes, but all final DU data are produced from the <100-mesh fraction. (See also Attachment B, EBA Field Work Flow).

QAPP Worksheet #18: Sampling Locations and Methods
(UFP-QAPP Manual Section 3.1.1 and 3.1.2)
(EPA 2106-G-05 Section 2.3.1 and 2.3.2)

Details of how the sampling locations will be selected are provided in Attachments A and B (the field work flow plans). The table below provides a summary of sample characteristics.

Sampling Location / ID Number	Matrix	Depth (inches bgs)	Analytical Group	Number of Samples**	Sampling SOP References	Comments
Lead Sweetening Area*	Soil	0 to 6 inches	Pb	Minimum: 30 Maximum: 60	TIIB Sample Collection 1.6 (under development)	Approximately 40 samples will be SU (9 to 16 point composites) and 8 - 20 will be DU (30 point composites)
	Soil	6 to 24 inches	Pb	Minimum: 30 Maximum: 60	TIIB Sample Collection 1.6 (under development)	Approximately 40 samples will be SU (9 to 16 point composites) and 0 to 20 will be DU (30 point composites)
Ethyl Blending Area*	Soil	0 to 6 inches	Pb	Minimum: 18 Maximum: 30	TIIB Sample Collection 1.6 (under development)	At a minimum, there will be 12 SU samples (9 to 16 point composites) and 6 30-pt DU-IS samples
	Soil	6 to 24 inches	Pb	Minimum: 18 Maximum: 30	TIIB Sample Collection 1.6 (under development)	At a minimum, there will be 12 SU samples (9 to 16 point composites) and 6 30-pt DU-IS samples

*Sample ID numbers will be assigned at the time of sampling according to the Sample ID strategy outlined in Attachment E. A map showing the approximate sampling locations for the LSA is provided in Worksheet 17, Figure 17-2.

** Because of the adaptive nature of this sampling plan, the exact number of samples cannot be determined prior to mobilization. It will depend on the extent of contamination determined in real-time based on the field laboratory results. Not all samples collected may require sample processing and analysis; however, "superfluous" samples will be retained until project completion in case data gaps are revealed that they can fill.

QAPP Worksheet #19 & 30: Sample Containers, Preservation, and Hold Times
(UFP-QAPP Manual Section 3.1.2.2)

(EPA 2106-G-05 Section 2.3.2)

Laboratory: Analysis conducted in the field at the site with 2 XRF instruments
Back-up Laboratory: None
Sample Delivery Method: Samples will be hand delivered to the analysis team from the field sampling team.

Analyte or Analyte Group	Matrix	Method/ SOP	Accreditation Expiration Date	Container(s) (number, size & type per sample)	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround
XRF Metals (Pb)	Soil	**Sample Prep:** XRF Procedure 1.2 "ISM Sample Processing" **XRF Analysis:** XRF Procedure 1.3 "Taking XRF Readings on Soil in Plastic Bags"	Not Applicable	One gallon size plastic bags for each sample collected in the field (approx. 1-4 kg)[1]	None. Samples are held at room temperature and stored in file boxes when not in use.	2 years	2 years	Real-time XRF data validation includes real-time evaluation of QC data. Final data package requires reconciliation of RTeX forms, logbooks & instrument files to check for clerical errors before study data are considered "validated."
Lab analysis of select samples	Soil	TBD, if RPM decides lab analysis is needed based on the outcome of this field effort.		Information provided in Wilcox Oil Procedure 1.4[2]	None.	2 years	2 years	Variable.

Notes: [1] These are the requirements for samples collected in the field. Samples will be processed, re-weighed and re-bagged in approved plastic bags for the XRF analysis.

[2] If the RPM determines that certain samples should be submitted for comparability analysis (such as ICP or IVBA), information on the collection, processing and containerizing of these laboratory samples is provided in *Wilcox Oil XRF Procedure 1.4. Subsampling and Analysis for Comparability Assessments.*

QAPP Worksheet #20: Field QC Summary
(UFP-QAPP Section 3.1.1 and 3.1.2)
(EPA 2106-G-05 Section 2.3.5)

Matrix	Analyte/ Analytical Group	Field Samples	Field Replicates	Matrix Spikes	Matrix Spike Duplicates	Field Blanks	Equip't Blanks	Trip Blanks	XRF Instrument Blank	XRF LCS controls	XRF Replicate Bag Readings	Total # analyses
Soil	XRF for Pb only	Variable, depending on sample result and decision logic	SUs: 2 side-by-side (couplets) DUs: 3 field DU-IS replicates at selected DUs	0	0	0	0	0	SiO_2 blank run at least 2 times per day	Group of 3 LCSs run & plotted at least 4 times per day	At least 4 readings per bag analysis; more if indicate by bag statistics	Variable, depending on sample result and decision logic

QAPP Worksheet #21: Field SOPs
(UFP-QAPP Manual Section 3.1.2)
(EPA 2106-G-05 Section 2.3.2)

Reference Number	Title, Revision Date and / or Number	Originating Organization	Topic	Modified for Project Work? (Y/N)	Comments
Wilcox Sample Collection Procedure 1.6	Incremental Sample Collection Using a Thin Walled Driven Probe/Corer	USEPA OSRTI TIIB	Collecting incremental composite soil samples up to 24 inches with thin walled tube (core/probe) sampler	No	Procedure is specific to Wilcox Oil: The 24-inch core will be separated into 0-6" and 6-24" segments.
Wilcox Sampling Equipment Decontamination Procedure 1.7	Non-disposable Equipment Decontamination	ICF	Cleaning of core samplers	No	

QAPP Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection
(UFP-QAPP Manual Section 3.1.2.4)
(EPA 2106-G-05 Section 2.3.6)

Field Equipment	Calibration Activity	Maint. Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP Reference
Digital balance	Per manual	Per manual	Per manual	Per manual	Daily, if used	Per manual	Per manual	Field Sample Lead / Field Lab Lead	User Manual
Sieve Shaker	NA	Per Manual	NA	Per manual	Per manual	NA	Per manual	Field Lab Lead	User Manual

QAPP Worksheet #23: Project SOPs and Procedures
(UFP-QAPP Manual Section 3.2.1)
(EPA 2106-G-05 Section 2.3.4)

Reference Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
Wilcox Oil XRF Procedure 1.1	Operation of the TIIB Niton XL3t GOLDD Ultra (Under Development)	Definitive	Metals	Niton XL3t GOLDD Ultra XRF	USEPA OSRTI TIIB	No
Wilcox Oil XRF Procedure 1.2	ISM-XRF Sample Processing v1.0 April 2017	Definitive	Sample Prep	Not Applicable	USEPA OSRTI TIIB	No
Wilcox Oil Procedure 1.3	Taking XRF Readings on Soil in Plastic Bags v1.0 April 2017	Definitive	XRF Metals	XRF (general)	USEPA OSRTI TIIB	No
Wilcox Oil Procedure 1.7	Non-disposable Equipment Decontamination	N/A	N/A	N/A	N/A	No
Wilcox Oil XRF Procedure 2.0	The Instrument Evaluation Period v1.0 April 2017	Definitive	XRF Metals	XRF (general)	USEPA OSRTI TIIB	No
Wilcox Oil XRF Procedure 2.1	Assessing Instrument Precision and Bias v1.0 April 2017	Definitive	XRF Metals	XRF (general)	USEPA OSRTI TIIB	No
Wilcox Oil XRF Procedure 2.2	Creating and Using XRF Control Charts v1.0April 2017	Definitive	XRF Metals	XRF (general)	USEPA OSRTI TIIB	No
Wilcox Oil XRF Procedure 2.3	Using the XRF Bag Checker and Calculator Tool v1.0 April 2017	Definitive	XRF Metals	XRF (general)	USEPA OSRTI TIIB	No
Wilcox Oil XRF Procedure 2.4	Subsampling and Analysis for Comparability Assessments v1.1 August 2017	Definitive	XRF Metals	XRF (general)	USEPA OSRTI TIIB	No

QAPP Worksheet #24: Analytical Instrument Calibration
(UFP-QAPP Manual Section 3.2.2)
(EPA 2106-G-05 Section 2.3.6)

Instrument	Calibration Procedure	Calibration Range	Frequency	Acceptance Criteria	Corrective Action (CA)	Title/position responsible for Corrective Action	SOP Reference
Niton XL3t Gold Ultra	Energy calibration check at startup. Concentration calibration status determined prior to project, and is not altered during the project.	Concentration calibration check performed from 11 to 5600 ppm	Energy calibration done each time instrument is started	Calibration check not significantly different from previous check as established by silica blank and LCSs	Troubleshoot instrument if LCSs indicate a change in instrument calibration (see XRF Procedure 02.1.2: Creating and Using XRF Control Charts)	Deana Crumbling	TIIB XRF Procedures 02.1.1 and 02.1.2
Olympus X-5000	As above	As above	As above	As above	As above	Henry Gerard	As above

QAPP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection
(UFP-QAPP Manual Section 3.2.3)

(EPA 2106-G-05 Section 2.3.6)

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Title/position responsible for corrective action	Reference
Niton XRF (TIIB, S/N 92959)	Clean dust from outside of instrument and stand	Run the 99.995% SiO_2 blank	Detector membrane for dust and tears	Upon receipt of the instrument; and 2 x per day during operation	SiO_2 blank is ND for Pb	Blow dust off membrane; Replace membrane if torn	Deana Crumbling or operator	Thermo Fisher Scientific Niton XL3 XRF User's Guide v. 7-0-1 Nov 2010
Olympus X-5000	As above	As above	As above	As above	As above	As above	Henry Gerard or operator	Olympus manual

QAPP Worksheet #26 & 27: Sample Handling, Custody, and Disposal
(**UFP-QAPP Manual Section 3.3**)
(**EPA 2106-G-05 Section 2.3.3**)

Sampling Organization: EPA OSRTI TIIB with ERT and ICF contractor support

Laboratory: On Site Field Laboratory

Method of sample delivery (shipper/carrier):   Hand delivered from field team

Number of days from reporting until sample disposal: TBD

Activity	Organization and title or position of person responsible for the activity	SOP reference
Sample labeling	Field Sample Team Leader and designated field team samplers	None. Sample labeling scheme described in Attachment E
Chain-of-custody form completion	Field Sample Team Leader	None.
Packaging	No special packaging is required as samples will be analyzed in the field laboratory.	Not Applicable
Shipping coordination	No shipping is required as samples will be analyzed in the field laboratory.	Not Applicable
Sample receipt, inspection, & log-in	Field Laboratory Team Leader	None for field samples. For processed samples to be analyzed by XRF: *Wilcox Oil XRF Procedure 01.3: Taking XRF Readings On Soil In Plastic Bags*
Sample custody and storage	Field Laboratory Team Leader	None. Requirements described in addendum below.
Sample disposal	EPA Region 6, in consultation with OSRTI Project Manager	Some samples will be disposed back to the site. Other samples may be retained for additional analyses at a later time; will be determined in consultation with Region 6.

QAPP Worksheet #28: Analytical Quality Control and Corrective Action
(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)
(EPA 2106-G-05 Section 2.3.5)

Matrix: Soil
Analytical Group: Metals
Analytical Method/SOP: Metals by XRF (see Worksheet #23 for specific Procedures)

Matrix: Soil			**Concentration Level:** Low to High			
Analytical Group: Metals			**Analytical Method/ SOP Reference:**			
QC Procedures	**Frequency / Number**	**Method / SOP QC Acceptance Criteria**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Check XRF Calibration	Every 6 months	Curve consistent with initial curve with good linearity	Send to Manufacturer for servicing and recalibration	Deana Crumbling	Bias & Precision	Linearity up to 5600 ppm; slope (for at least 5 CRMs <1500) = 1.0+/-0.1; intercept = 0 +/- 10; r^2 >0.99
Blank analysis with 99.995% silica	If XRF used outside: before each LCS run (4 X per day) If XRF used inside: 2 x per day	Target analytes are ND	Check blank membrane for dust; check XRF platform for dust.	XRF Analyst	Accuracy/Bias	No cross-contamination; target analyte should be non-detect
Instrument Replicate analysis	Once per day, unless indicated by an unusual matrix	See XRF Procedure 2.1	Evaluate spectrum for sample-specific matrix interference	XRF Analyst	Precision/ Interference	Record in the RTeX form; instrument precision should be similar to other samples
Run Lab Control Samples (LCSs)	4 times per day (AM start up; before lunch, after lunch, and at end of day	Adapted from Westgard Rules for QC acceptance	Check cup membrane for damage or dust	XRF Analyst	Bias & Precision	See XRF Procedure 02.2 (3 concentration levels)
Paper Control Charting of LCSs (Target analytes)	100% - each chart unique to instrument, analyte, CRM, and scan time	See XRF Procedure 02.2	See XRF Procedure 02.2	XRF Analyst	Accuracy/Bias	Readings within +/- 2 SD control limits; Corrective actions as directed in SOP
Read-Bag Interference checks	Once per lot of plastic bags using 3 conc. levels of LCSs	Bag Check Calculator indicates no significant interference	See XRF Procedure 02.3	XRF Analyst	Bias & Precision	Bag Check Calculator indicates no significant interference for target analyte

QAPP Worksheet #29: Project Documents and Records
(UFP-QAPP Manual Section 3.5.1)
(EPA 2106-G-05 Section 2.2.8)

Record	Generation	Verification	Storage location/archival
Sample Collection and Field Records			
Field logbook or data collection sheets	Field Sample Team Leader	TIIB Project Manager	TIIB Project File
Photo documentation	Field Sample Team Leader	TIIB Project Manager	ICF Project File
Daily quality control reports	Field Sample Team Leader	TIIB Project Manager	TIIB Project File
Chain of custody	Field Sample Team Leader	TIIB Project Manager	TIIB Project File
XRF Analysis Documents and Records			
XRF-RTeX (sample analysis) forms	XRF Analyst or clerical assistant	Field Lab Team Leader	Long term XRF data storage on CD ROM @ TIIB
XRF instrument data files (.ndt files)	XRF Analyst	Field Lab Team Leader	Long term XRF data storage on CD ROM @ TIIB
Original XRF data downloads (Excel files)	XRF Analyst	Field Lab Team Leader	Long term XRF data storage on CD ROM @ TIIB
Logbooks	XRF Analyst	Field Lab Team Leader	TIIB Project File
Sample preparation logs (weights, dry times)	Sample Prep Staff	Field Lab Team Leader	TIIB Project File
Sample storage report	Field Lab Team Leader	TIIB Project Manager	TIIB Project File
Project and Data Assessment Documents and Records			
Field and lab audit checklists	Field Lab Team Leader	TIIB Project Manager	TIIB Project File
Corrective action forms	Field Lab Team Leader	TIIB Project Manager	TIIB Project File
Project Reports and Interim Work Products			
Data reduction and visualization work-products (e.g., ProUCL, Surfer, analysis)	All	TIIB Project Manager or Technical Lead	TIIB Project File
Meeting notes and collaborative work products/tools	All	TIIB Project Manager	TIIB Project File
Project reports	All	TIIB Project Manager	TIIB Project File

XRF-RTeX form = XRF Real-Time Excel = the Excel spreadsheet form used to evaluate & run statistics on XRF data from samples in real-time.

TIIB Project File may include upload to ftp or EPA SharePoint sites

QAPP Worksheet #31, 32, and 33
Planned Project Assessments Table

Assessments:

Assessment Type	Responsible Party & Organization	Number/Frequency	Estimated Dates	Assessment Deliverable	Deliverable due date
Readiness Review	EPA TIIB (Matt Jefferson) or designee	One assessment two weeks prior to mobilization	September 2017 (TBD)	Readiness Review Memorandum and Checklist (ICF)	24 hours following assessment
Field Sampling Surveillance	EPA TIIB (Matt Jefferson) or designee	One each on first day of sampling area for SU and DU sampling	September 2017 (TBD)	Notes and Team Debrief	24 hours following assessment
Soil Preparation Surveillance	EPA TIIB (Matt Jefferson) or designee	Once for each sampling area	September 2017 (TBD)	Notes and Team Debrief	24 hours following assessment
XRF Analysis Surveillance	EPA TIIB (Matt Jefferson) or designee	Once at beginning of project	September 2017 (TBD)	Notes and Team Debrief	24 hours following assessment

Assessment Response and Corrective Action:

Assessment Type	Responsibility for responding to assessment findings	Assessment Response Documentation	Timeframe for Response	Responsibility for Implementing Corrective Action	Responsible for monitoring Corrective Action implementation
Readiness Review	Field Sample Team Leader	Readiness Review Corrective Action Response	24 hours from receipt of Readiness Review Memorandum	As directed by PM	EPA TIIB (Matt Jefferson)
Field Sampling TSA	Field Sample Team Leader	Field Sampling Corrective Action Response	24 hours from receipt of Memorandum	Field Task Leader	EPA TIIB (Matt Jefferson)
Soil Preparation Surveillance	Field Lab Team Leader	On-site Analytical Corrective Action Response	24 hours from debrief	Field Lab Team Leader	EPA TIIB (Matt Jefferson)
XRF Analysis Surveillance	Field Lab Team Leader	On-site Analytical Corrective Action Response	24 hours from debrief	Field Lab Team Leader	EPA TIIB (Matt Jefferson)

Note: Any member of the field team may issue a stop work order if safety issues arise. In addition, the EPA project manager will be responsible for any other stop work orders.

QAPP WORKSHEET #34

Data Verification and Validation Inputs

Item	Description	Verification (completeness)	Validation (conformance to specifications)
Planning Documents/Records			
1	Approved QAPP and Field Sampling Plan	X	
2	Contract/Scope of Work	X	
4	Field Sampling SOPs	X	
5	Field Laboratory SOPs	X	
Field Records			
6	Field logbooks	X	X
7	Sample location files (GPS, or other)	X	X
8	Chain-of-Custody Forms	N/A	N/A
9	Sampling diagrams/surveys	X	X
10	Relevant Correspondence	X	X
11	Change orders/deviations	X	X
12	Field surveillance reports	X	X
13	Field corrective action reports	X	X
Sample Prep and XRF Data			
14	Master Sample Log in Sheet - chronology (i.e. dates and times of receipt, preparation, & analysis)	X	X
15	Internal laboratory chain-of-custody, weight/tare/duration sheets for drying, sieve shaker report sheets, sieve fraction weight sheets	X	X
16	Control Charts	X	X
17	Disaggregation Completeness Analysis Sheet	X	X
18	Plastic Bag Checker Analysis	X	X
19	Non-conformance log	X	X
20	XRF Logbooks	X	
21	XRF RTeX forms (sample measurement results and statistics)	X	X

Item	Description	Verification (completeness)	Validation (conformance to specifications)
22	CRM Standards Traceability	X	X
23	XRF Instrument evaluation period records	X	X
24	Raw data download from XRF instruments (Niton .ndt files with spectra & photos; Olympus spectra output; Excel files with numerical instrument output)	X	
25	Corrective action reports	X	X
26	Communication records	X	X

QAPP WORKSHEET #35
Data Verification Procedures

This worksheet documents procedures that will be used to verify project data. Data verification is a completeness check to confirm that all required activities were conducted, all specified records are present, and the contents of the records are complete. Data verification (as defined in the UFP-QAPP Manual, 2005) will be performed for the XRF data by after-project reconciliation of the field log books (sample collection records and XRF logbook), XRF raw data files, and RTeX sample-recording forms to ensure completeness, accuracy and preservation of all data-related records.

Records Reviewed	Requirement Documents	Process Description	Responsible Person, Organization
Field Documentation	QAPP and related Procedures (SOPs)	Verify that records are present and complete for each day of field activities. Verify that all planned samples including field QC samples were collected and that sample collection locations are documented. Verify that meteorological data were provided for each day of field activities. Verify that changes/exceptions are documented and were reported in accordance with requirements. Verify that any required field monitoring was performed and results are documented.	Daily – Field Team Leader (Matt Jefferson) At conclusion of field activities - Project QA Manager (Deana Crumbling)
Chain-of-custody forms	QAPP	None, unless off-site sample analysis requested by RPM	Deana Crumbling
Field Laboratory Data	QAPP	Verify that the field laboratory deliverables contain all records specified in the QAPP. Compare the RTeX forms to verify that results were provided for all collected samples. Review the log book to ensure all QC exceptions are described. Check for evidence that any required notifications were provided to project personnel as specified in the QAPP. Verify that necessary signatures and dates are present.	Project Technical Lead (Deana Crumbling)
Audit Reports, Corrective Action Reports	QAPP	No audits planned.	

QAPP WORKSHEET #36
Data Validation Procedures

This worksheet documents procedures that will be used to validate project data. Data validation is an analyte and sample-specific process for evaluating compliance with contract requirements, methods/SOPs, and Measurement Performance Criteria.

No samples from this study are planned for analysis by a fixed labs or submitted for EPA Standard Methods analysis; therefore, no 3rd party data validation will be performed. If the RPM decides on site to request lab analysis on selected sample(s), the RPM will be responsible for getting those data validated.

Data validation (as defined in the UFP-QAPP Manual, 2005) will be performed for the XRF data by

- pre-project evaluation of the XRF calibration status and performance for the target analyte,

- real-time evaluation of LCS results before and after running samples,

- real-time assessment of sample representativeness by evaluation of field replicates and sample analysis replicates, and

- after field work review/reconciliation of logbooks, RTeX forms and instrument data files.

The Data Usability Assessment described in Worksheet #37 will be applied to the XRF data generated for this project.

3rd Party Data Validator: None

Analytical Group/Method:	NA	NA
Data deliverable requirements:	NA	NA
Analytical specifications:	NA	NA
Measurement performance criteria:	NA	NA
Percent of data packages to be validated:	NA	NA
Percent of raw data reviewed:	NA	NA
Percent of results to be recalculated:	NA	NA
Validation procedure:	NA	NA
Validation code (*see attached table):	NA	NA
Electronic validation program/version:	NA	NA

QAPP WORKSHEET #37
Usability Assessment

This worksheet documents procedures that will be used to perform the data usability assessment. In this project, data usability will be largely assessed in real-time during data collection activities through real-time statistical evaluation of sample data and monitoring of QC checks. Real-time evaluation allows immediate identification of sampling or analytical problems that could affect data usability. A final usability assessment will performed after the field portion of the project is completed using the outputs from data verification and data validation. The final assessment will be described in the project report.

The Key personnel responsible for performing the usability assessment is Deana Crumbling (USEPA) with assistance from ICF support staff. Other project staff will participate in eh data usability assessment during the various project phases including:

- EPA Region 6 Remedial Project Manager – Katrina Higgins-Coltrain
- OSRTI TIIB Project Manager – Matt Jefferson
- Risk Assessor – Phil Turner (EPA Region 6)
- Field Sampling Leader/Geologist- Jim Rice

Summarize the data usability assessment process including statistics, equations, and computer algorithms that will be used to analyze the data:

Step 1	**Review the project's objectives and sampling design**
	An Adaptive Sampling Plan approach is applied for this investigation to identify the area where soils between the surface and 2 feet contain lead at concentrations greater than 200 ppm. As part of that process sample results, generated in real time, are used to define the approximate location of the 200 ppm boundaries at both sites. Decision Logic Diagrams, provided in the Work Plan, are used to guide the selection of sample locations. Geostatistical analysis using a kriging method to develop and iso-concentration contours (EVS©), is used to define the boundary line.
Step 2	**Review the data verification and data validation outputs**
	XRF Data • The XRF data generated during field mobilization will be validated as usable via real-time QC activities that include monitoring instrument and operator performance. This will be accomplished by real-time charting of LCS QC and real-time verification that instrument duplicate QC results are acceptable (See the relevant SOPs for more information). If QC results are not acceptable, real-time trouble-shooting and correction of any problems will be performed before data are reported. If necessary, sample data not bounded by in-control LCSs will be rerun after corrective action is successful. ○ All reported XRF data are required to be bounded by in-control QC results. Thus, no reported XRF data should be rejected at a later time due to QC non-conformance. • During field work, the Field Team supervisor will perform spot-checks to ensure field staff are following XRF operation and XRF data entry procedures. Any observed deviations from procedures will be addressed by the

	field supervisor or designee, and if needed, staff will be retrained.
	○ LCS control charts (these are paper) will be inspected by the supervisor to ensure real-time charting is being performed and control chart documentation is adequate. Completed paper control charts and their accompanying "Notes/Troubleshooting" sheets will be stored in a safe location and scanned into electronic files as soon as possible.
	○ Past and current Instrument Duplicate QC Calculator files will be checked for complete entry information. Completed files (these are electronic Excel files) should be properly stored and backed up. This may involve password protection to avoid accidental changes to a completed file.
	○ Previous and current DU-Bag Concentration Calculators (electronic RTeX Excel spreadsheets) will be inspected to ensure that all required spreadsheet inputs are filled out, and that statistical significance was attained for each final bag sample concentration result. Completed files should be properly stored and backed up. This may involve password protection to avoid accidental changes.
	○ Written entries in field notebooks covering the relevant time periods will be scanned into electronic files that are stored with the relevant, completed spreadsheet files so that meta information is readily accessible.
Step 3	**Verify the assumptions of the selected statistical method** The geostatistical analysis used to generate iso-concentration contours at the LSA uses a kriging methodology and is dependent on the spatial density of data points (field sample locations). The geostatistical analysis will include a qualitative (and if available, quantitative) assessment of spatial coverage to ensure the 200 ppm boundary line is adequately constrained. Real-time assessment of modeling uncertainty will be addressed by placement of additional samples in indicated locations. One assumption that will be tested and assessed during real time data evaluation is that concentration is highest at the source and decreases in a predictable pattern away from the source. Minor deviations from assumptions are not critical to statistical analysis and data interpretation. Overall measurement error will be assessed by measuring the amount of sampling error attributable to soil heterogeneity within a DU by taking three independent replicate (triplicate) DU samples. • It is critical that these field replicates be independent, which means that they are collected as 3 separate, but identical increment collections. The only difference is the increment layout, which must cover the same area, but be offset so that two increments do not fall on the exact same spot. • Ideally, the increments from all 3 field replicates will evenly cover the DU. • Each sample must have the same number of increments, and to the extent possible, the same increment mass. • Overall measurement error is calculated as the %RSD for the 3 replicate field samples. • DU samples having only a single field sample will have their UCLs predicted using a consensus %RSD derived from replicated DUs using the procedure described in Attachment A, Section 3, Item 3.

	Sufficient DUs should be selected for field replication QC so that there is confidence that either 1) all DUs have similar soil heterogeneity, or 2) soil heterogeneity varies with depth and/or by the site soil type. Field heterogeneity (as %RSD) is determined by adding the subsampling and analytical variances together and subtracting that sum from the total variance. The square root of the variance is the standard deviation (SD), which is divided by the DU mean to obtain the RSD. XRF analysis of a sample bag relies on developing a statistically robust measurement of the 95% Upper Confidence Limit (UCL) on the mean concentration of Pb in the sample, based on multiple XRF measurements of the sample. The spreadsheet used for calculation of the 95 UCL applies a well-established statistical test (Student t-test or Chebyshev test) and does not need to be verified. It should be possible to use the Student's t confidence limits, since a normal data distribution is expected for repeated XRF readings on a sieved, non-segregated bag. However, if high within-bag heterogeneity persists after corrective action efforts, it may be necessary to use the Chebyshev UCL and LCL.
Step 4	**Implement the statistical methods** • As described in Step 3 above, the EVS©, geostatistical package will be applied for determination of the initial 200 ppm boundary line at LSA. A qualitative analysis of tolerance can be perfumed by plotting the residuals at sample points. • Evaluate statistical error in DU data and calculate or predict (using the procedure in Attachment A, Section 3, Item 3) the 95% UCL for confirmatory DUs. The ideal tolerance for field triplicates is 20% RSD. If that is exceeded, replicate sample precision is still acceptable if the 95% t-UCL is < 200 ppm. • Analytical error (precision) will be quantified during analysis by using multiple XRF measurement of the sample. The tolerance for uncertainty in analytical measurements varies with particle size. For particle size fractions <60-mesh, the tolerance is 15% RSD. For <10-mesh samples, the tolerance is 30% RSD. When decision-making relies on a single bag concentration (such as decisions based on individual SU results), the 1-sided 95% Students-t UCL will be used as the estimate of the sample bag mean (the bag UCL is calculated in real-time by the RTeX form). Excessive statistical uncertainty in bag decisions due to an elevated UCL will be resolved by increasing the number of XRF readings for the bag, and/or remixing the bag. • Instrument error is provided by the XRF instrument for each reading and can be improved in many cases by using a longer read time, if necessary. It is anticipated that the XRF read time for this project can be reduced to 15-20 sec because of the analyte (Pb), expected concentrations (greater than 100 ppm), and the nature of many of the decisions (semi-quantitative decisions on coarse particle size fractions of SU samples).
Step 5	**Document data usability and draw conclusions** The written deliverable will include a data usability summary report which can be in the form of text and/or a table identifying if the data can be used as intended, considering implications of deviations and corrective actions, based on the aforementioned data quality indicators. Performance of the sampling design and limitations on data use will also be presented. The final deliverable will update the conceptual site model and document conclusions. Permanent data records in the form of RTeX forms, raw XRF output spreadsheet files, QC records and Excel data analysis and calculation files, and field and XRF logbooks will be provided.

Attachment A – LSA Field Work Flow Plan

Meet the night before to tentatively assign next day's field tasks

Anticipated work crews:
- obstacle clearing
- sample collection
- sample processing/XRF analysis
- geostatistical modeling/field communications

Preliminary Stage:
- Flag center 2 highest concentration areas per the "blue ERT kriged map" (concentrations >300K ppm), using ERT's GPS coordinates (see end of this file for the "blue map" figure)
- Flag planned transects (#1 to #16) at ERT's 1000 ppm and the 200-500 ppm line contour lines as given on the "green ERT kriged map" shown in the figure in QAPP Worksheet #17. (see end of this file for the "green map" figure)
    - The 1000 ppm line is the outer edge of the yellow-green concentration band,
    - The 500 ppm line appears to be the outer edge of the green band, and
    - The modeled 200 ppm line is the pink line.

Section 1: SU Stage #1 (SU Testing and Optimization)
The initial test of SU performance (described also in Worksheet #12 in the "Representativeness" section) will use SU side-by-side couplets, each having 9 increments over a 2 x 2 sq.ft. area (in 3 rows of 3) for a 0-6" depth interval only for one of the couplets, and both the 0-6" and 6-24" intervals for the other couplet member (to evaluate the contaminant pattern at depth). Three to four sets of couplets will span a high to low concentration range along an easily accessible transect, as illustrated by the figure below. If SU performance (agreement between the two 0-6" interval samples) is inadequate (refer to Worksheet #12 in the "Representativeness" section), the area will be enlarged and the number of increments will be increased.

Item 1. Transect Line for SU testing
- Choose a transect line that appears to cover the full concentration range while being easily accessible by the field crew.
- Test-SUs are 4 sets of side-by-side couplets (couplets should be 1-2 ft. apart as measured between adjacent sides); collect both the 0-6" and 6-24" intervals for each couplet, and place each interval into a separate bag.
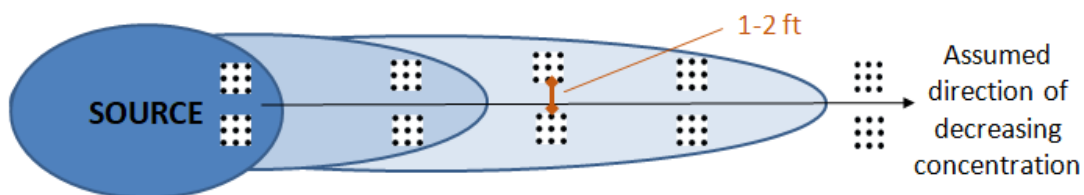
First trial transect using series of nine-point incremental samples (SUs) away from the source to test the reliability of SU configurations

- Place Test-SU Couplet #1 on the innermost SU location of test transect (refer to the "ERT green map" at the end of this file)
    - Examine the soil surface and samples from the innermost SU couplet for crystalline materials that could be Pb compounds
- Test-SU Couplets #2, 3 (and possibly 4) are placed farther out along the test transect.

- The last Test-SU Couplet should be near the pink line (modeled 200 ppm line) on the "green map"
- Test-SU samples should be delivered to the processing/XRF crew.
    - Initially, only the 0-6" interval samples are processed. The 6-24" samples are set aside until later.
    - Disaggregate and sieve 0-6" samples to <10-mesh if needed to remove vegetation or large bits of non-representative material
    - The analytical portion will be placed in a large "read bag"
    - XRF sample readings on the Test-SUs may be taken using a 15-sec XRF read time (unless the concentration is <100 ppm, in which case a 30-sec read time should be used)
    - Use the SU RTeX form for sample bag mean and statistics to control for within-sample heterogeneity
    - XRF analysis of the 0-6" interval samples for all 3 or 4 Test-SU couplets should be completed before judging the adequacy of the SU configuration
        - Using the sample bag mean as the result, is the information provided by the members of couplets consistent for deciding whether a trend exists?
        - If yes, complete analysis of the 6-24" Test-SU samples. Is the trend information consistent in the subsurface samples (i.e., if there is Pb >200 ppm in any subsurface samples, is there a pattern of decreasing concentration with distance from the center of the LSA)?
        - Using the sample bag mean as the result, evaluate the concentration difference between the members of couplets using the Excel SU Couplet Evaluator (see screen shot below):
            - Is the lowest concentration member within 40% of the highest concentration member?
            - Ex. If the highest concentration member is 600 ppm, is the concentration for the lower member of the couplet > 360 ppm?

SU Couplet Evaluator

SU Identifier	Highest couplet conc	Lowest couplet conc	minus 40% of highest	Is the lowest within 40%?
A	600	450	360	yes
B	400	200	240	no

Item 2. If the 3x3 increment/2x2 ft^2 area configuration for the 0-6" interval is inadequate (indications of concentration trend is inconsistent among any of the 4 couplets or between-couplets), use the degree of inconsistency to decide whether to go to the 3x4 increment/3x3 ft^2 or 4x4 increment/4x4 ft^2 configuration.

2 x 2 sq.ft. SU increment configuration

3 x 3 sq.ft. SU increment configuration

4 x 4 sq.ft. SU increment configuration
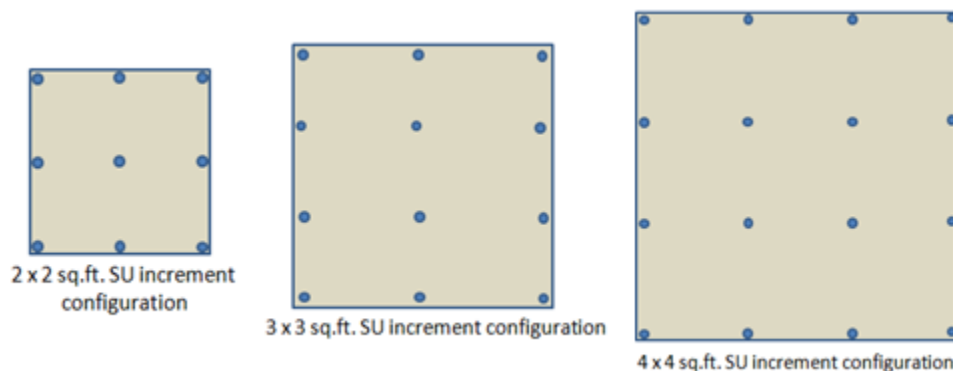
- If initial SU performance is _markedly_ inadequate, the SU configuration will be changed to 4 x 4 sq.ft. area and 16 increments (4 rows of 4).

- If performance is only <u>marginally</u> inadequate, the area will be enlarged to 3 x 3 sq.ft. and 12 increments (3 rows of 4)
- Another 3 to 4 sets of test couplets should be collected using the new configuration to confirm adequate performance.
    - Repeat testing with the new SU configuration along the test transect, but offset slightly from the previous SU sampling locations
    - Evaluate agreement between couplet members as described before
    - Also evaluate the degree of agreement between the first and second SU sets
        - This gives a sense of the degree of short-scale heterogeneity (lateral noise), which influences the ability to reliably detect a concentration trend
- The 6-24" interval samples from an unsuccessful SU configuration can be discarded back to the site.

Section 2: SU Stage #2 (Delineation data collection)
After optimizing the SU configuration, SU sampling along transects will begin near the outer borders identified as being near 200 ppm by the kriged *in situ* XRF Pb results from the 2015 field work (see ERT-produced figures at the end of this file).

_____

Depth consideration
If the Test-SU transect found that subsurface contamination (i.e., Pb >200 ppm in the 6-24" depth interval) was not present (except perhaps at the very highest concentrations in the LSA center), SU sampling at the outer boundary does not need to include the 6-24" interval.

- However, if there is any doubt about where subsurface contamination ends, SU samples should include the deeper interval in locations where there is uncertainty about the presence of subsurface Pb.
- If any subsurface Pb levels >200 ppm are found, the areal extent of the contaminated 6-24" interval must be delineated using SUs and geostatistical modeling.
- Since the 0-6" interval is likely to be much higher than the 6-24" interval, SUs sampled solely to determine the extent of subsurface contamination do not require processing and analysis of the 0-6" interval.

_____

Item 1. If there were only 3 Test-SU couplets performed in Section 1, collect a couplet at the first SU location of the first delineation transect as additional confirmation of the selected configuration.

- If all SU results from the first SU location exceed 200 ppm (as measured by the 95% UCL on the XRF bag analysis), the next SU location will be further out along the transect.
    - The subsequent SUs can be singlets.
- If all SU results from the first location are less than 200 ppm, the next SU will be located further in toward the center of the LSA.
    - The subsequent SUs can be singlets.
- If the SUs are not giving consistent information, another SU configuration should be considered.
- When the putative 200 ppm boundary has been bracketed, place a second SU next to the decisive SU (to make a couplet) to confirm that conclusion
    - This step of confirming a decision indicated by one SU by coupling with a second SU should be performed for >75% of the SU transects.

Item 2. <u>SU singlets</u> will be used along the rest of the transects, except when confirming the final SU of a transect.

- Sieving to 10-mesh will be done as needed:
    - LSA soils that are nearly all sand and finer grain sizes, with no stones or with little aggregated material present, do not need to be sieved since nearly all material would pass through.

- XRF sample readings may be taken using a 15-sec XRF read time (unless the concentration is <100 ppm, in which case a 30-sec read time should be used)

Item 3. Place the 1st SU of subsequent transects on the 200-400 ppm contour line shown in the ERT "green map" (if there is confidence in the contouring accuracy of the map) or place it near the suspected 200 ppm line indicated by on-going data collection.
- Based on the sample results, move inward or outward along the transect to place the next SU. The goal is to have an SU above 200 ppm and at least 1 SU below 200 ppm along each transect.
  - o Distance to the next SU placement along a transect will use professional judgment based on the actual SU concentrations found and any pattern developing as data collection moves forward.
  - o Inward placement of SUs will stop once there is confidence that concentrations further along the transect will all be >200 ppm Pb.

Item 4. When a group of SU transect data has been completed, provide the data to the geostatistical operator for concentration contour modeling.
- Additional SUs/SU couplets or whole transects may be placed between existing transects to increase spatial coverage where professional judgment finds excessive uncertainty in the geostatistical modeling.

Item 5. When convenient for the sample collection crew, SU samples (both 0-6" and 6-24") should be collected from the two locations flagged as the highest *in situ* concentrations ERT found (refer to the "green map").
- Both the surface and subsurface samples at each of the 2 locations should be examined visually for Pb crystallization
- Both the surface and subsurface samples at each location should be analyzed to evaluate the relationship between surface and subsurface Pb concentrations.
- If the subsurface Pb concentrations are <200 ppm at both high concentration locations, and other subsurface analyses performed to that point also show subsurface concentrations <200 ppm, processing and analysis of other subsurface samples may be put on hold, depending on the consistency of subsurface sample results:
  - o The consistency of Pb concentrations in the subsurface samples will be examined to determine whether enough subsurface data is available to determine the Pb concentration in the subsurface interval outside of any subsurface "hot spot" >200 ppm. This information is needed for mathematically combining data from the 0-6" and 6-24" intervals to generate 0-24" interval concentrations for ecological risk assessment purposes.
    - ▪ Consistency in Pb concentrations throughout all or most of the subsurface indicates that fewer subsurface samples will require analysis
    - ▪ Inconsistent subsurface Pb concentrations will require analysis of more subsurface samples.
    - ▪ Again, geostatistical modeling may assist to determine when sufficient data has been generated to support decisions.

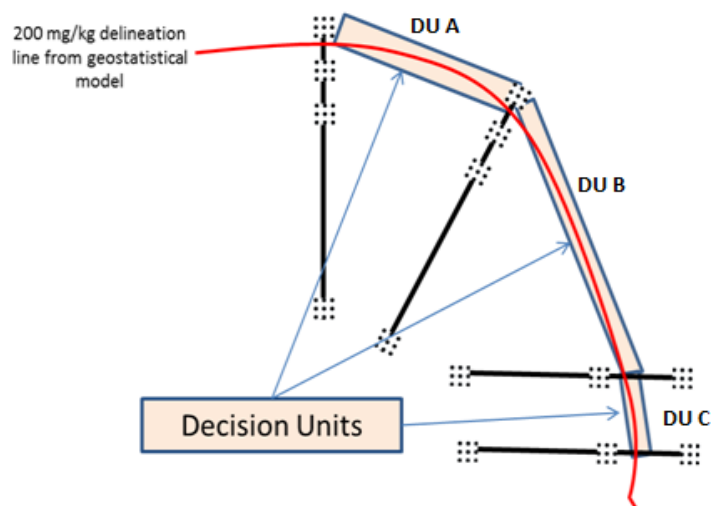**Section 3: DU Stage (Surface boundary confirmation incremental sampling)**
The following strategy will significantly reduce the sample collection and XRF workloads by avoiding taking incremental sample replicates on every boundary DU, yet will ensure that false negative decision errors are avoided. The target frequency for triplicate samples in border DUs is 20% (3 DUs at a minimum). The strategy outlined below should ensure that those DUs needing replication in order to limit decision uncertainty will be preferentially selected into those 20%.

Introductory Note: The first reading of the decision-making and work flow process described below may seem complicated, however it is actually straight-forward in implementation.

- The benefit of the following decision-making strategy is the time and labor saved by reducing the number of field samples requiring collection in potentially difficult soil or vegetation conditions, while maintaining a measurably high degree of decision confidence.
- The following mathematical strategy is possible for the following reasons:
  - o Highly accurate quantitation of each DU's Pb concentration is not required, since the data need only demonstrate at high confidence that border DUs meet the boundary condition of 200 ppm or less.
  - o Therefore, decision uncertainty can be managed through mathematical/statistical mechanisms, which can be performed with just the typing of a few numbers into an Excel calculator, which is much faster and easier than field sample collection and analysis.

Item 1. DU placement: When modeling of the SU data is judged to have produced a sufficiently confident estimate of the 200 ppm contour line, band-shaped DUs (about 10 ft. wide) will be used to confirm the border sections of the surgical cleanup area

- A border confirmation DU may be placed over the 200 ppm contour line bridging transects (as in the figure to the right), or may be positioned with the DU's inner edge on the contour line.
  - o The choice of DU position will rest with professional judgment based on the level of confidence in the modeled 200 ppm contour line.
- Border confirmation DUs will have areas ranging between 1000 and 6000 square feet.
- Since a maximum of 16 transects are anticipated, a maximum of 15 border DUs are anticipated.
  - o The field QC goal for DU data is to have triplicate replication in at least 20% (1/5th) of the boundary confirmation DUs.
  - o Therefore, a minimum of 3 border DUs need triplicate incremental samples (ISs), although it is likely there will be more.
- Border DUs will be sampled with 30 increments per IS.
- Pb concentrations in the subsurface soil this far from the LSA center are anticipated to be consistently well below 200 ppm (as demonstrated the SU data).
  - o If this holds true, boundary confirmation DUs will sample only the 0-6" surface soil interval.
  - o If this does not hold true, border DUs may require sampling of both the surface (0-6") and subsurface (6-24") intervals.

Item 2. DU sample processing: All final DU decisions are based on the Pb content in the <100-mesh particle fraction. Samples must be sufficiently disaggregated to produce a sufficiently representative <100-mech sample.

- If soil conditions are such that disaggregation is difficult, and if it is found that DU "step-outs" are frequently required, the following procedure can be used to save time and labor:
  - o Sieve several DU samples to <10-mesh initially and analyze
  - o Re-sieve the samples to <100-mesh and analyze
  - o Compare the results for the <10-mesh and <100-mesh particle fractions
    - ▪ If the concentration difference between the two fractions is insignificant, or if the concentration of the <10-mesh fraction is greater than that of the <100-mesh fraction, the 10-mesh fraction can be used as an intermediate value until the final DU footprint is achieved

Item 3. DU representativeness QC strategy: This strategy will allow reduction of the sampling workload by using a single DU-IS sample to conservatively estimate the DU concentration for first testing of a border DU. In this way, 95% statistical decision confidence can be maintained without the need for field triplicates for every border DU. The following describes how this QC strategy will operate:

- The initial 2 border DUs will have triplicate 30-point ISs, and for each the following test will be applied:
  - If all 3 IS results are <200 ppm, and the 1-sided 95% t-UCL for those 3 results is <200 ppm, that border DU is acceptable.
  - If any of the 3 IS results are >200 ppm, or the UCL is >200 ppm, that border DU is unacceptable. That DU's footprint will be moved outward and the triplicate sampling process repeated.
- The first 2 acceptable DUs count toward the minimum of 3 replicated DUs for the project.
- Once 2 acceptable border DUs are established, the variability data from these 2 DUs will be used to establish a QC Criterion for each future DU (DU-X). The DU-specific QC Criterion will determine the acceptability of each DU for representing the 200 ppm border for that section of the boundary. The QC Criterion is calculated using the following steps:
  - From the first 2 acceptable DUs, the RSD for each set of triplicates will be calculated, and those 2 RSDs averaged (using the relative variance as the averaging intermediate). In other parts of the QAPP, this value is called a "consensus %RSD" to avoid confusion over what an "average" %RSD is.
    - The Excel formula for the average RSD is =sqrt(sumsq(value1,value2)/2)
- This average ("consensus") RSD will be used to calculate a DU-specific SD that will be used to predict a UCL for any DU having only a single IS collected from it.
  - Letting DU-X IS = DU-X's single IS result, the following is the Excel equation that will be used to calculate the predicted UCL (which will serve as the DU-specific QC Criterion):
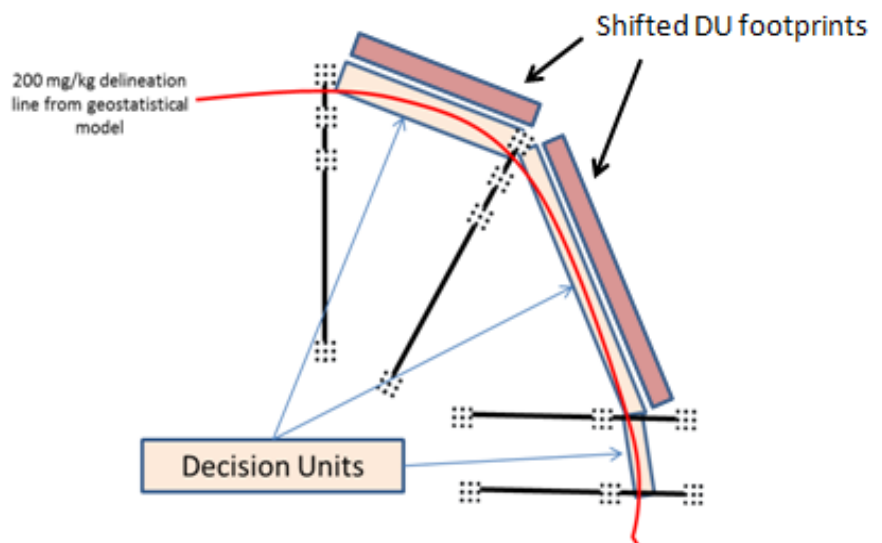
    DU-X QC Criterion = DU-X IS + (TINV(0.1,2)*(DU-X IS*ave RSD)/sqrt(6))

    [Note: The square root of 6 is used because the RSD value was derived from 2 sets of DU-IS triplicates. Although the group of 6 are not truly independent in the context of the sqrt(n) term, it is expected that the 2 DUs will have nearly the same concentration, and the violation of independence will have minimal effect for the intended use of the predicted UCL as a QC acceptance criterion.]

  - Using the QC Criterion is a mechanism to account for within-DU data variability while reducing the sampling workload by using a single DU-IS sample to conservatively estimate the DU concentration.
- An Excel calculator has been set up to assist with all these calculations ("Border DU Criterion calculator"), see the "Calculation of Criterion Value's RSD for Predicting DU UCLs" figure at end of Section 5.

Item 4. A DU with only a single IS result is considered acceptable if its DU QC Criterion value is 200 ppm or less; any DU with an IS result >200 ppm is automatically judged unacceptable.

Item 5. Unacceptable DUs will have a step-out DU placed and sampled, as shown in the figure:

Item 6. If an un-replicated DU has an IS result well below 200 ppm, but the predicted UCL/QC Criterion value is only slightly >200 ppm, 2 additional DU replicates can be collected to see if the actual 95% UCL is less than the predicted. If the actual UCL on the mean is <200 ppm, then that DU is acceptable.
- Extra sampling of a DU may be favored over shifting the DU footprint if difficult field conditions (such as dense shrubby undergrowth and trees) are present in the step-out locations.

Item 7. To meet the overall DU QC frequency stated in Worksheet #12, a total of 3 DUs must be replicated.
- The 3rd DU to be replicated would ideally be chosen from a border segment that has relatively higher uncertainty in the geostatistical modeling.
- If sufficient modeling uncertainty exists, additional DUs should be replicated.
- Confirm that RSDs from the 3rd (or more) replicated DUs are similar to the RSDs from the first 2 border DUs.
    - If the new RSDs are higher than those from the first 2 DUs, recalculate the average RSD and use it to recalculate predicted UCLs for previous un-replicated DUs.
    - If previously acceptable DUs are now unacceptable, either use a step-out DU, or collect 2 additional field replicates and calculate the actual UCL on the DU mean.
    - Use the new RSD for calculating the QC Criterion for future un-replicated DUs.

Item 8. At the end of the DU process, at a minimum there will be a cleanup footprint defined by the 200 ppm boundary as proven by the ring of DU border segments that cover the 0-6" depth.

Section 4: Subsurface delineation
- From indications provided by the 2015 Pb data, it is anticipated that the area of >200 ppm contamination in the 0-6" interval will overlay and extend well beyond that in the 6-24" interval.
- Contamination in the 6-24" interval may be sufficiently delineated by the SUs collected thus far. If not, additional subsurface SUs may be required to complete geostatistical modeling of the 200 ppm boundary in the 6-24" layer.

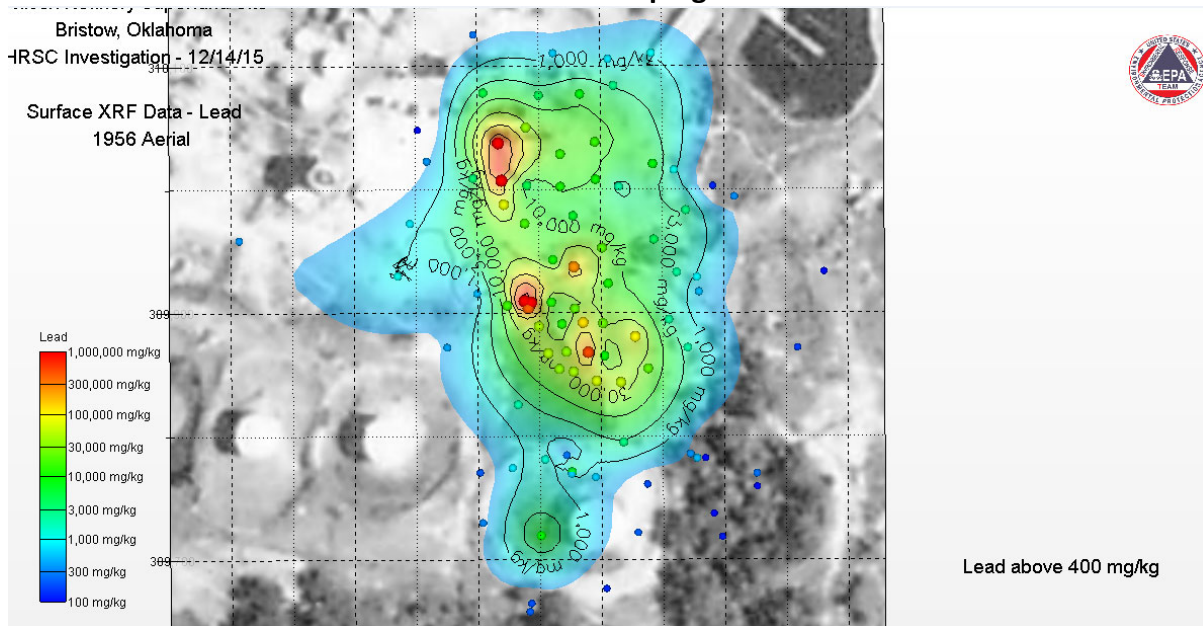**Section 5: LSA wrap-up to produce materials for use by the RPM**
- Flag boundaries
- Take sufficient GPS readings and distance measurements to produce accurate maps
- Use GIS or other spatial modeling to generate estimation of the volume of soil with Pb concentrations >200 ppm for the surface and subsurface intervals.
- Where applicable, mathematically combine the 0-6" and 6-24" interval data to produce an exposure point concentration for the 0-24" interval.

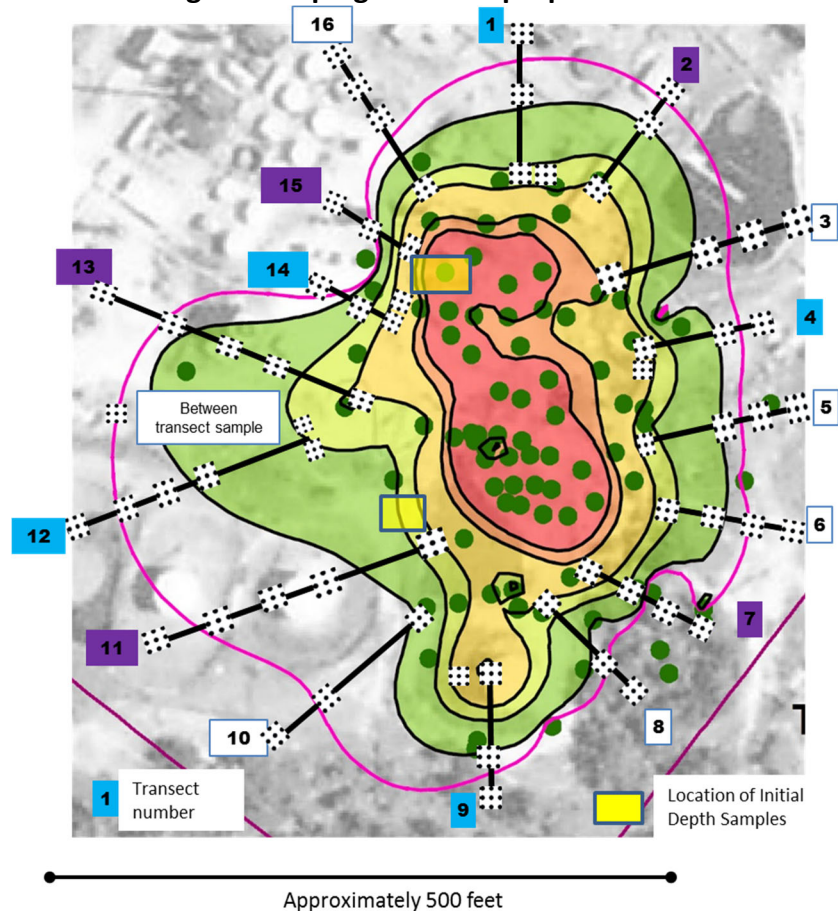### Calculation of Criterion Value's RSD for Predicting DU UCLs

Calculation of Criterion Value's RSD				**Criterion Value calculation & record-keeping for each singlet DU-IS**				
								DU Co
Enter the following values for DUs A & B:	DU A bag conc	DU B bag conc		Enter the following value for single DUs:	DU ID	DU-IS initial result	Criterion Value	
DU-X-IS-Rep1	150	130			DU C	175	**195.8**	lat
DU-X-IS-Rep2	175	145						long
DU-X-IS-Rep3	180	160			DU D			
DU X's mean conc =	168.3	145.0						
DU X's SD =	16.1	15.0			DU E			
DU X's mean RSD =	0.095	0.103						
DU X's mean %RSD =	9.5%	10.3%			DU F			
DU X's 95% t-UCL =	195.4	170.3						
					DU G			
relative variance for DU X =	0.0091	0.0107						
averaged relative variance for DUs A & B =	0.0099							
average RSD for DUs A & B =	**0.100**							

ERT's "blue map figure"

Bristow, Oklahoma
HRSC Investigation - 12/14/15

Surface XRF Data - Lead
1956 Aerial

Lead
1,000,000 mg/kg
300,000 mg/kg
100,000 mg/kg
30,000 mg/kg
10,000 mg/kg
3,000 mg/kg
1,000 mg/kg
300 mg/kg
100 mg/kg

Lead above 400 mg/kg

ERT's "green map figure" with proposed transects

Between transect sample

Transect number

Location of Initial Depth Samples

Approximately 500 feet

Graphical summary of LSA field work plan

```
LSA start
   │
   ▼
┌──────────┐        ┌─────────────────┐
│Transect 1│        │ ▢ = SU work     │
│   SUs    │        │ ▢ = DU work     │
└──────────┘        └─────────────────┘
   │          ┌──────────────┐
   │◄─────────│ SU Pb data   │
   ▼          │   & QC       │
              └──────────────┘
◇ Adequate SU      ▲
  configuration? ──No──► ┌─────────────┐
   │                     │ Modify SU   │
  Yes                    │configuration│
   ▼                     │and recollect│
┌──────────────┐         └─────────────┘
│ Sample SU at │
│ outer end of │◄─────────────────────┐
│ new transect │                      │
└──────────────┘                      │
   │                                  │
   ▼                  ┌─────────────────────┐
◇ SU Pb <200          │Sample new SUs outward│
  ppm?  ──No──────────►│along transect until Pb│
   │                  │ slightly <200 ppm    │
  Yes                 └─────────────────────┘
   ▼                          │
┌──────────────┐      ┌───────────────┐      ◇ Colocated SU
│Sample new SUs│      │ Place & sample│──No──► ~ same conc?
│inward along  │─────►│ colocated SU  │
│transect until│      └───────────────┘
│Pb slightly   │
│<200 ppm      │
└──────────────┘
```

(Flowchart continued — right side)

```
┌────────────────────────┐
│ GPS LSA 200-ppm        │
│ boundary & calculate   │◄──Yes──┐
│ non-compliant volume   │        │
└────────────────────────┘        │
                        ◇ DU Pb UCLs
              ┌──No──────  slt <200 ppm?
              │                 ▲
    ┌──────────┐                │
    │ DU Pb    │                │
    │ UCL & QC │───────►        │
    └──────────┘         ┌──────────────┐
              │          │  Place &     │
              └─────────►│ sample DUs   │
                         └──────────────┘
                                ▲
                               Yes
                                │
                         ◇ Data sufficient
              No──────────  for SU geostat
              │              modeling?
       ┌────────────┐            │
       │Identify &  │           Yes
       │fill SU data│────────►   │
       │gaps        │      ┌───────────────┐
       └────────────┘      │ Go to next    │
                           │  transects    │◄──Yes── Colocated SU
                           └───────────────┘
```

Attachment B – EBA Field Work Flow Plan

Meet the night before to tentatively assign next day's field tasks

Anticipated work crews:
- obstacle clearing
- sample collection
- sample processing/XRF analysis
- geostatistical modeling/field communications

Preliminary Stage
- Flag location of highest hit in ERT's kriged map of 2015 XRF results (see end of this work flow)
- Inspect the 2 buildings and flag potential sources areas (PSAs) such as
    - Piping and valves,
    - Doorways
    - Outflows
    - Low-lying areas where spills could accumulate and infiltrate into the ground
    - Stained soil/distressed vegetation

Section 1: Investigation outside the buildings

Item 1. Collect one 9 to 16-point SU (using the default 3x3 over 2x2 sq.ft. configuration or the SU configuration optimized in the LSA work) at all flagged locations at both the 0-6" and 6-24" depth intervals.
- There should be at least 2 SUs on each side of the structures. If there are no PSAs on a side, place the SUs randomly.
- Process samples from both depth intervals by sieving through 10-mesh sieve; place the <2-mm particle size in a read bag and XRF while using the RTeX form to run statistics on multiple readings over the read bag
- Using the 1-sided 95% t-UCL from the RTeX form, identify any SU sample having a conservatively estimated mean Pb concentration >200 ppm. Such an SU is considered to be a "hot spot."
    - To evaluate the potential for hot spots, return to any >200 ppm location and collect additional SU samples (at both depths) near the original location and along the same side of the building
        - Place the additional SU samples such that their borders touch the borders of the first SU, and there is continual spatial coverage.
        - If all bordering SUs are <200 ppm, and if the SU configuration being used is larger than 4 sq.ft., cover the original SU (the high concentration area) with up to four 4-sq.ft. SUs to determine the actual size and location of the hot spot.
- *In situ* readings with hand-held XRF may also assist in this effort. See also the discussion in Attachment E (Sample Labeling and Identification), under "EBA SUs".
    - Ensure there a sufficiently dense pattern of *in situ* XRF readings to have confidence that the area is correctly identified
    - When initially taking readings in a particular SU, take at least 3 15-sec *in situ* readings in a single "spot" or "location" by moving the XRF slightly between readings. This is to control for particle effects that could cause non-representative individual readings.
        - After a pattern of *in situ* results has been established, SUs or locations that appear to show little variability (i.e., insignificant particle effects) do not need frequent replicate readings.
        - Replicate in situ readings can be reserved for verifying unusual or unexpected readings.
    - *In situ* field results are not entered into an RTeX form. The readings are used only as qualitative results to understand contaminant patterns within an SU or chase contamination that appears to be migrating.

- Detailed suggestions for how to record the results of *in situ* XRF readings in the field logbook and the XRF logbook appear in Attachment E, under "EBA SUs."
  - If the *in situ* results suggest that a concentration gradient away from a structure may be present, SU transects can be placed transects to characterize a possible trend prior to placing DUs.

Item 2. A hot spot will not be considered significant enough to warrant cleanup (and thus will not be flagged or mapped) if both of the following are true:

- There is only a single hot spot on a side of a structure, and
- That hot spot occurs only in the 0-6" interval, its area is no larger than 4 sq.ft., and its Pb concentration is no higher than 400 ppm.
- A hot spot smaller than 4 sq.ft. in the 6-24" interval may be considered significant if the circumstances suggest the detection may represent a subsurface migration pathway away from a spill source.
  - Subsurface detections of any size may require additional subsurface sampling to determine whether it represents a migration conduit transporting Pb off-site.
- Continue to place SU samples to bound >200 ppm areas, or in transects to define trends, until compliant soil is reached.
  - Flag the tentative outline of areas that will be targeted for cleanup
  - Place confirmation DUs (30 increments/sample and periodic triplicate samples) around the identified contaminated area(s) to
    - Document areas of compliant soil on each side of the buildings
    - Estimate the volume of soil needing removal
    - Determine concentrations for possible use in risk assessment

Item 3. Placing DUs: If all SU samples on a particular side of the building are <200 ppm in both depth intervals

- Place a 10-ft wide DU along that side of the building
- Collect 30 increments (3x10 or other configuration as indicated by SUs carved from the DUs) from both depth intervals (see figure below)
- Predicted UCLs may be employed (as described in the LSA Field Work Flow), however the variability term may be different from the variability determined for the LSA. If predicted UCLs are employed in the EBA, an EBA-specific variability term should be determined.
  - Collect triplicate ISs from the first 2 DUs to calculate their 95% UCLs, and to derive a consensus %RSD to use to predict UCL from single DU field samples for subsequent EBA DUs
  - The third EBA DU should have 3 replicate field samples as well to compare the actual against the predicted UCL using the consensus %RSD; modify the consensus %RSD using the third DU's %RSD if necessary.
  - This strategy is the same as that used in the LSA strategy (Attachment A, Section 3, Item 3)
- If DUs next to a structure have actual or predicted UCLs >200 ppm, add additional step-out DUs to determine the >200 ppm boundary around the structure
- See figure below
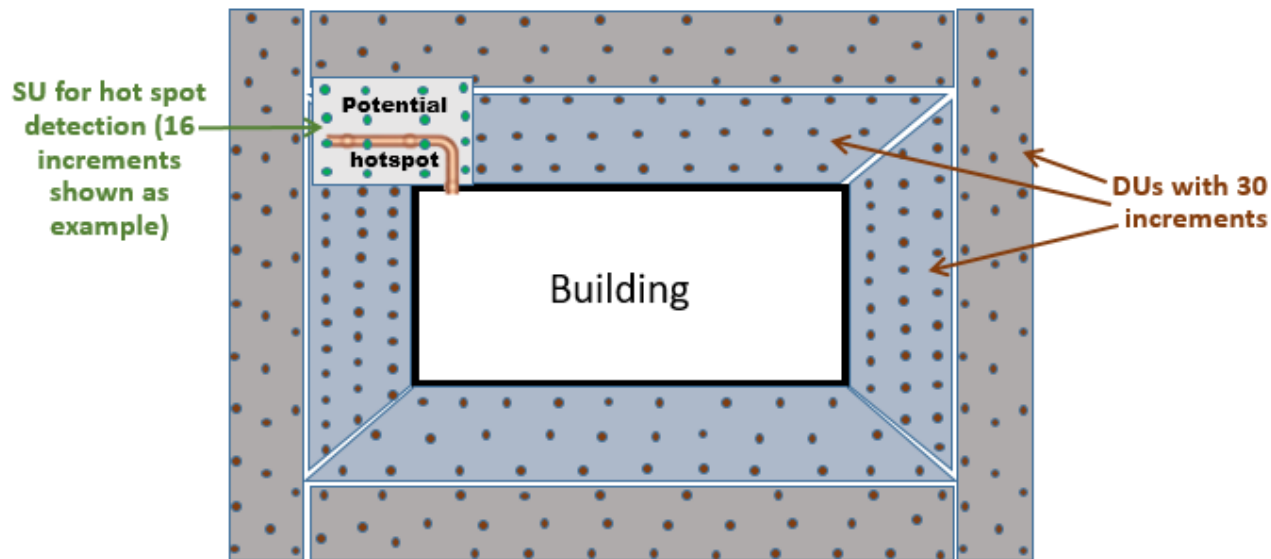
Item 4. <u>DU sample processing:</u> All final DU decisions are based on the Pb content in the <100-mesh particle fraction. Samples must be sufficiently disaggregated to produce a sufficiently representative <100-mech sample.

- If soil conditions are such that disaggregation is difficult, and if it is found that DU "step-outs" are frequently required, the following procedure can be used to save time and labor:
  - Sieve several DU samples to <10-mesh initially and analyze
  - Re-sieve the samples to <100-mesh and analyze
  - Compare the results for the <10-mesh and <100-mesh particle fractions
    - If the concentration difference between the two fractions is insignificant, or if the concentration of the <10-mesh fraction is greater than that of the <100-mesh fraction, the 10-mesh fraction can be used as an intermediate value until the final DU footprint is achieved

Item 5. Flag boundaries and take spatial measurements that will allow estimation of the volume of soil with Pb concentrations >200 ppm for possible removal.

- If unexpectedly high concentrations of Pb are found in the EBA, collect enough data to perform geostatistical modeling.
- Collect sufficient data at both depths to allow for risk assessment for each depth interval and in the combined (0-24" interval).

Section 2: Investigation inside the buildings

Item 1. Sample the dirt floor contained within the east building

- Place 1 or more DUs inside the building as circumstances indicate
- Sample from both depth intervals if possible
- Collect a single IS (30 increments) from each DU/depth interval
- Sieve and analyze the initial IS samples
  - If the east building DU samples are >200 ppm, designate the area for cleanup and additional ISs replicate samples are not required.
  - If the east building DU samples are <200 ppm, collect 2 additional replicate samples and calculate the 95% UCL for each DU/depth interval.

Item 2. Inspect the inside of the west building for soil or similar loose material that can be gathered into a read bag

- Collect and XRF those samples to determine whether inside construction materials might be contaminated with Pb.

Item 3. If desired by the RPM, an attempt may be made to use the Niton XRF in handheld mode to test materials in place (such as concrete and wood).

- Loose material may offer interference for the XRF, and the XRF results may not be reliable: this is a coarse screening only.
  - o Consider that the XRF is calibrated for a soil-type matrix, and may not perform equivalently for a material with a completely different matrix
  - o Check the spectra for misshapen peaks: peak shape different from those observed for a soil matrix indicate that any numerical readings are completely unreliable
- If peak shape appears acceptable, use multiple *in situ* shots over some area of a single material
  - o Determine and record the area over which the shots were made
  - o Calculate the average concentration for that area.

Section 3: EBA field wrap-up to produce materials for use by the RPM

- Flag boundaries
- Take sufficient GPS readings and distance measurements to produce accurate maps
- Use GIS or other spatial modeling to generate an estimate of the volume of soil with Pb concentrations >200 ppm for the surface and subsurface intervals.
- Where applicable, mathematically combine the 0-6" and 6-24" interval data to produce an exposure point concentration for the 0-24" interval.

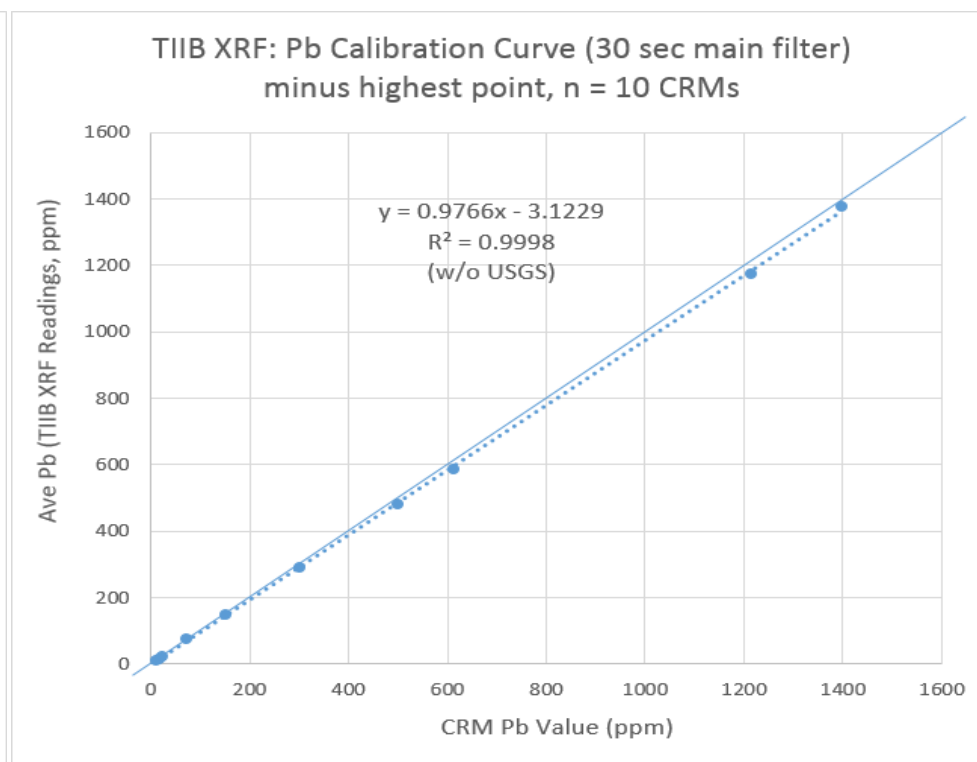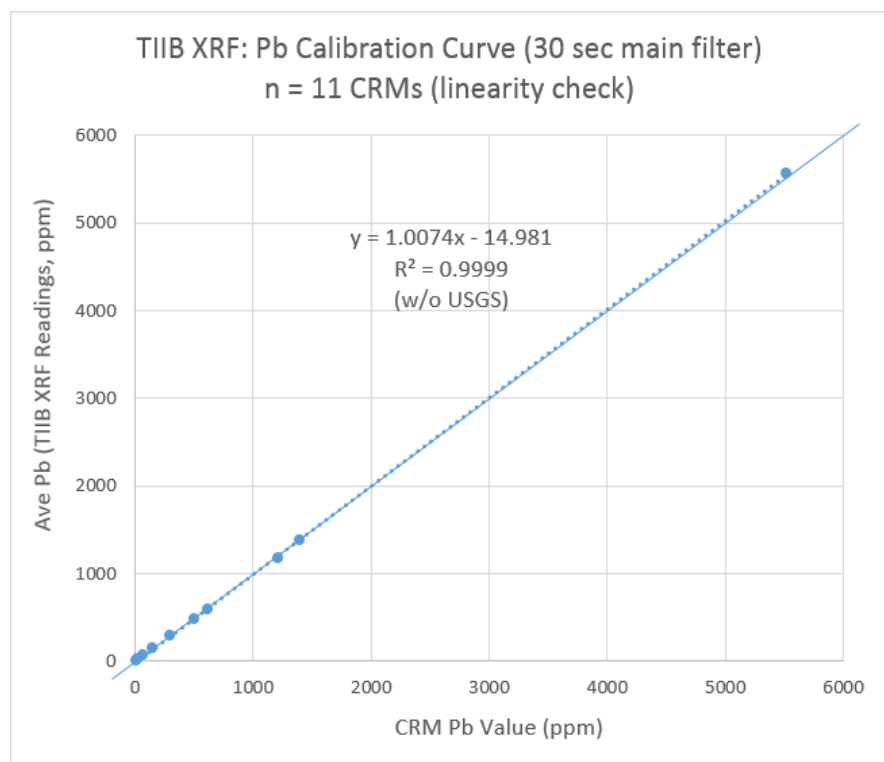**ERT's kriged EBA map**
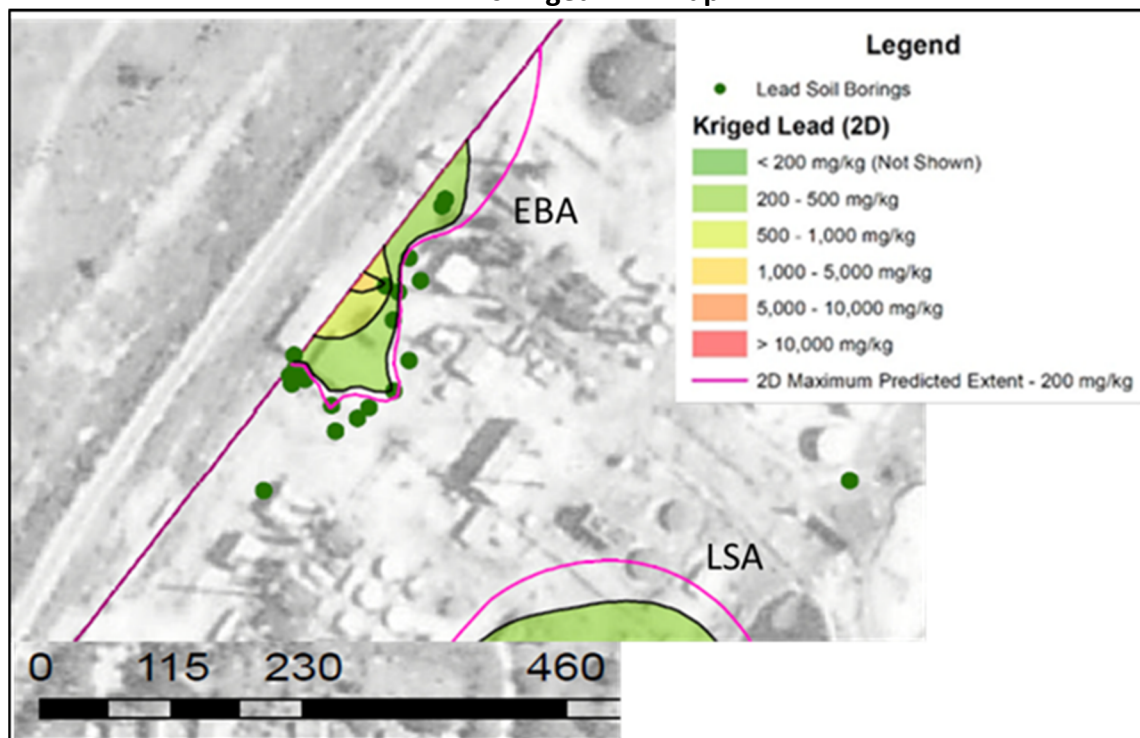
Graphical summary of EBA work plan
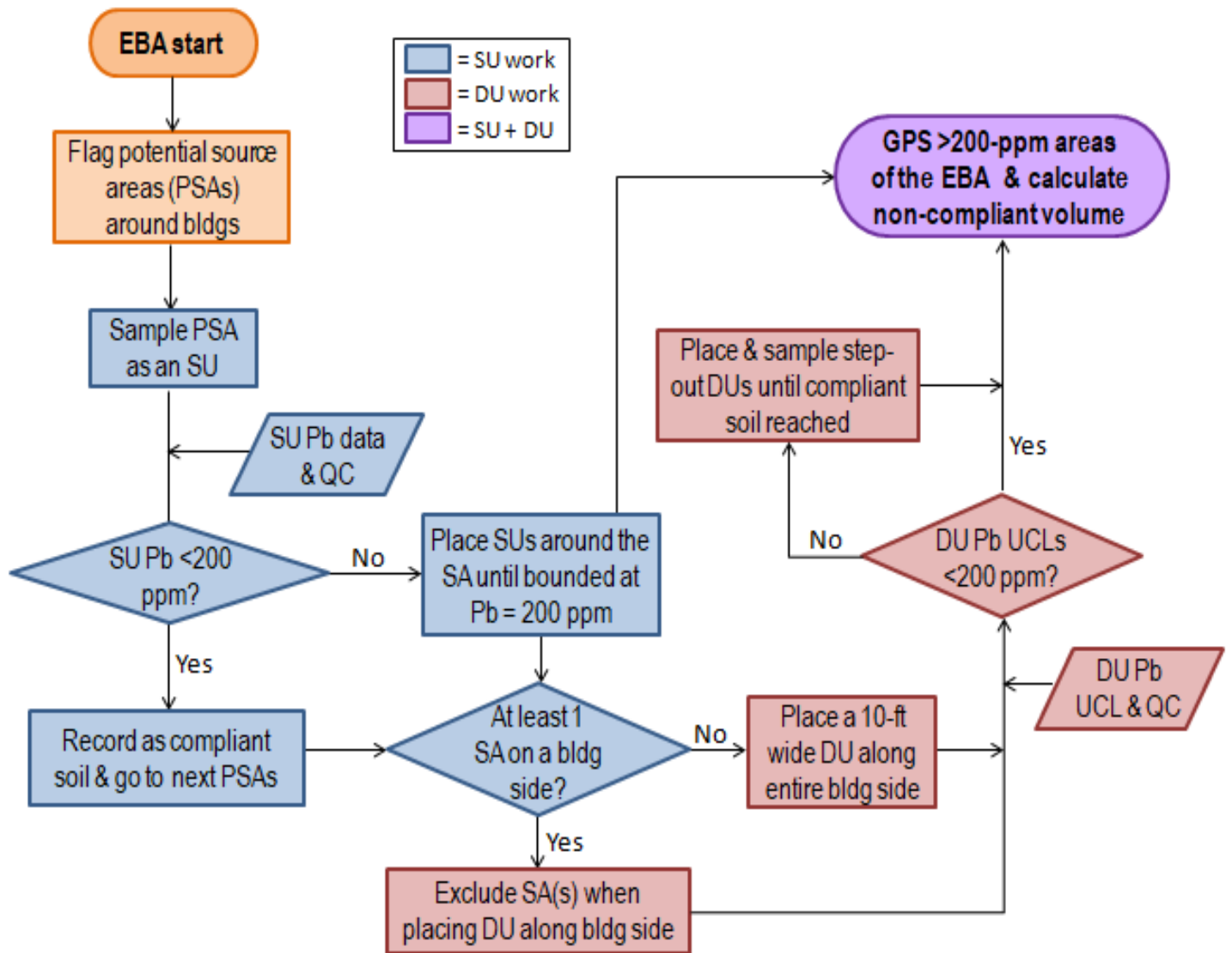
Attachment C: TIIB Niton XL3t XRF (S/N 92959) Operation Performance Measures

Calibration Status and Linearity Determination for Pb (current as of May 2017)

Linear to at least 5600 ppm Pb (see linearity check graph below left, which contains the extreme high data point)

Calibration curve parameters are determined from second curve (which lacks the extreme high data point that could bias regression parameters):

- Slope = 0.977; Y-intercept = -3.1 ppm; R^2 = 0.9998

TIIB XRF: Pb Calibration Curve (30 sec main filter) n = 11 CRMs (linearity check)

$y = 1.0074x - 14.981$
$R^2 = 0.9999$
(w/o USGS)

TIIB XRF: Pb Calibration Curve (30 sec main filter) minus highest point, n = 10 CRMs

$y = 0.9766x - 3.1229$
$R^2 = 0.9998$
(w/o USGS)

Niton instrument limit of detection (LOD) for Pb:

- 5.5 ppm (determined as 3 x signal/noise ratio, on a "clean" CRM with 11 ppm Pb concentration, and a 30-sec read time)
- 6.8 ppm (determined as 3 x signal/noise ratio, on a CRM with 24 ppm Pb concentration, and a 30-sec read time)

Niton instrument Quantitation Limit (QL) for Pb:

- 10 ppm (determined as the concentration at which the analytical %RSD = ~10 % in a "clean" matrix with a 30-sec read time)
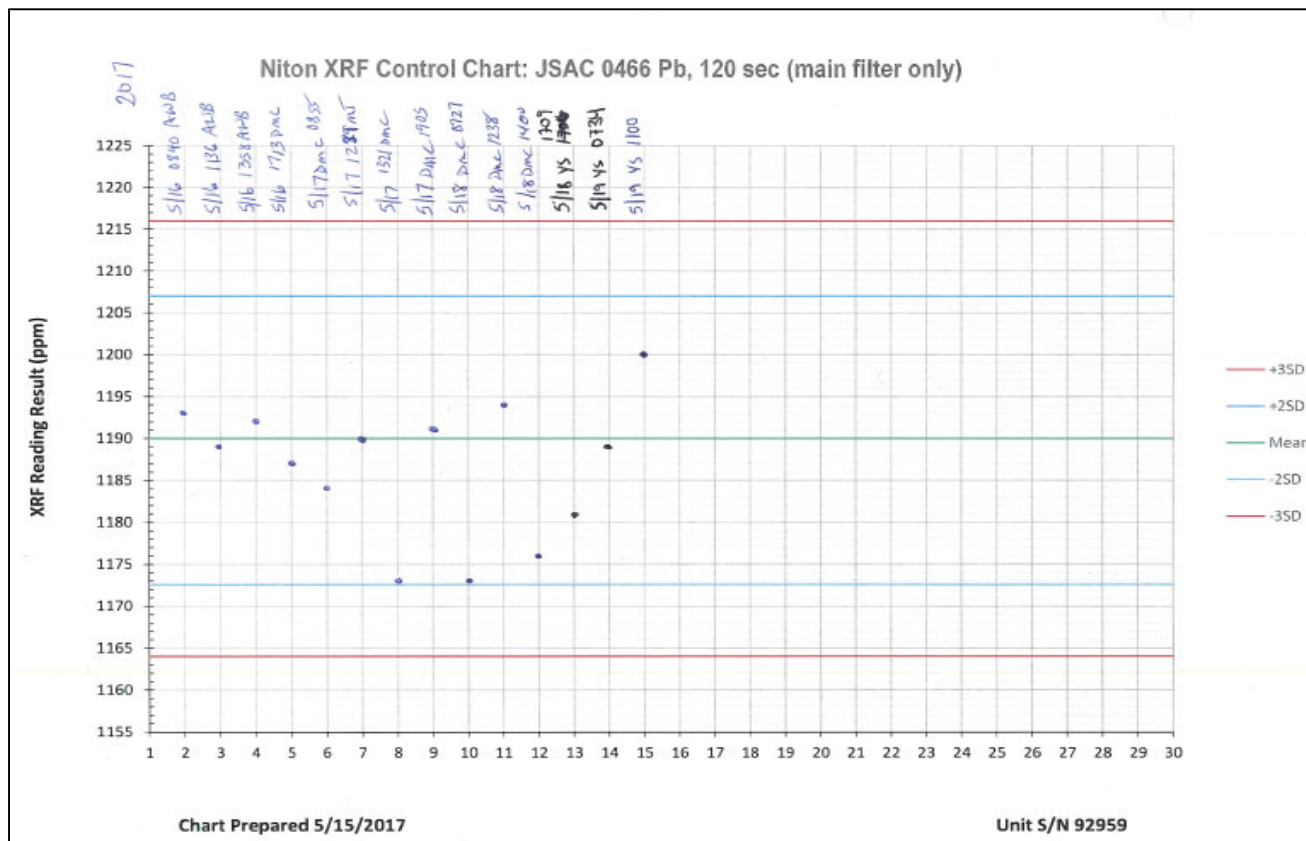
Niton instrument analytical precision:

- 8.8% RSD (on CRM with 11 ppm Pb and a 30-sec read time)
- 1.1% RSD (on CRM with 300 ppm Pb and a 30-sec read time)

Recent Niton control charts for 3 concentrations levels for Pb are shown below

Niton XRF Control Chart: JSAC 0466 Pb, 120 sec (main filter only)

Chart Prepared 5/15/2017   Unit S/N 92959
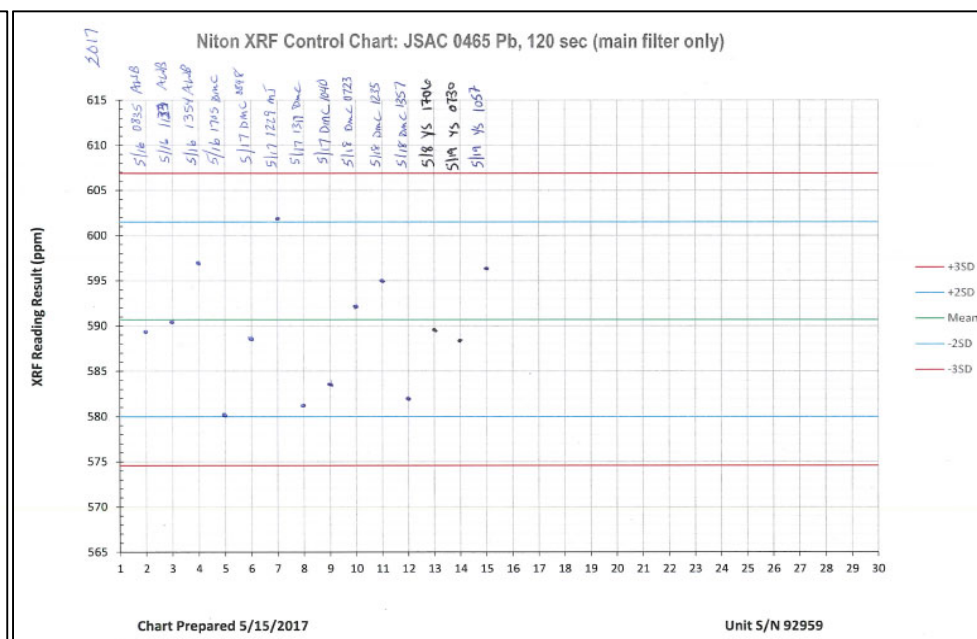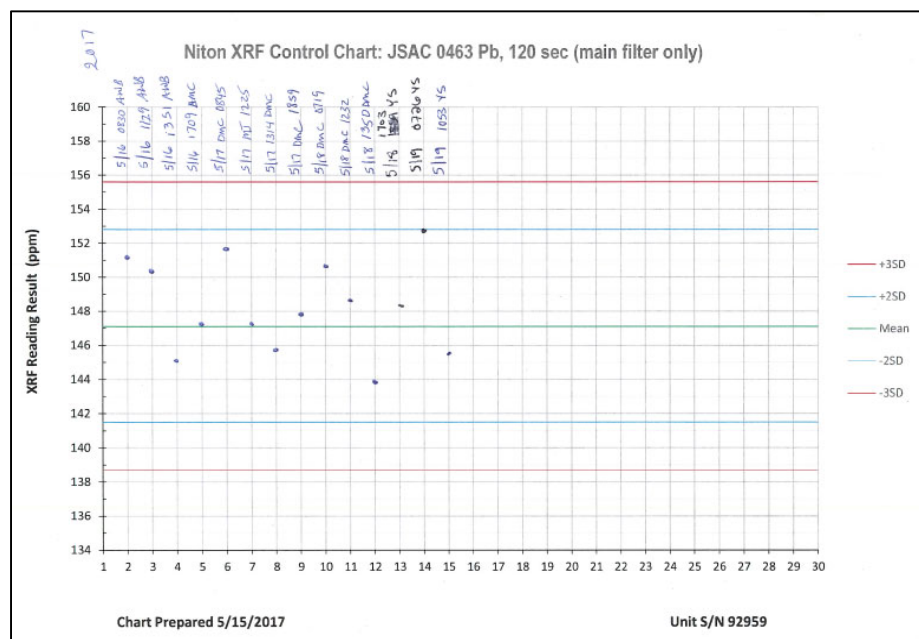
Niton LCS performance/control chart summaries (May 2017):

- JSAC 0463 (Low-level Pb) mean: 147 ppm (cert: 151.6 ppm = 97%); ±2 SD range: 142-153 ppm (±4.1%)
- JSAC 0465 (Mid-level Pb) mean: 591 ppm (cert: 612.4 ppm = 96%); ±2 SD range: 580-602 ppm (±1.9%)
- JSAC 0466 (High-level Pb) mean: 1190 ppm (cert: 1214 ppm = 98%); ±2 SD range: 1173-1207 ppm (±1.4%)

Attachment D: ERT Olympus X-5000 XRF (S/N 202309) Operation Performance Measures

Calibration Status and Linearity Determination for Pb (current as of May 2017)

Linear to at least 5600 ppm Pb (see linearity check graph below left, which contains the extreme high data point)

Calibration curve parameters are determined from second curve (which lacks the extreme high data point that could bias regression parameters):

- Slope = 0.983; Y-intercept = -4.6 ppm; R^2 = 0.9996
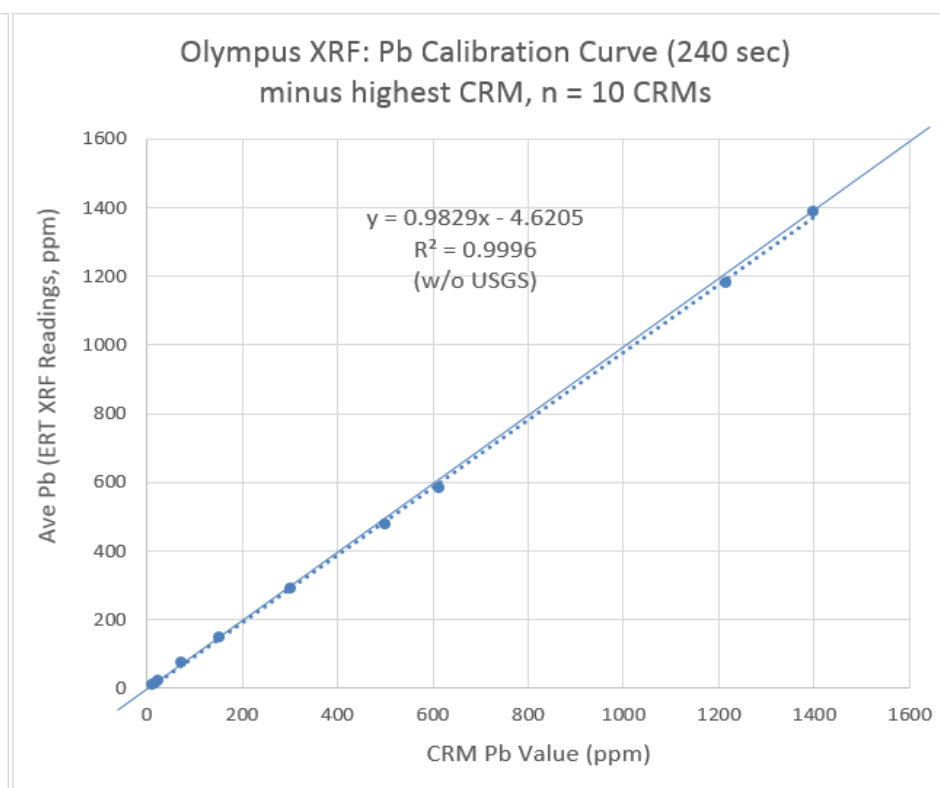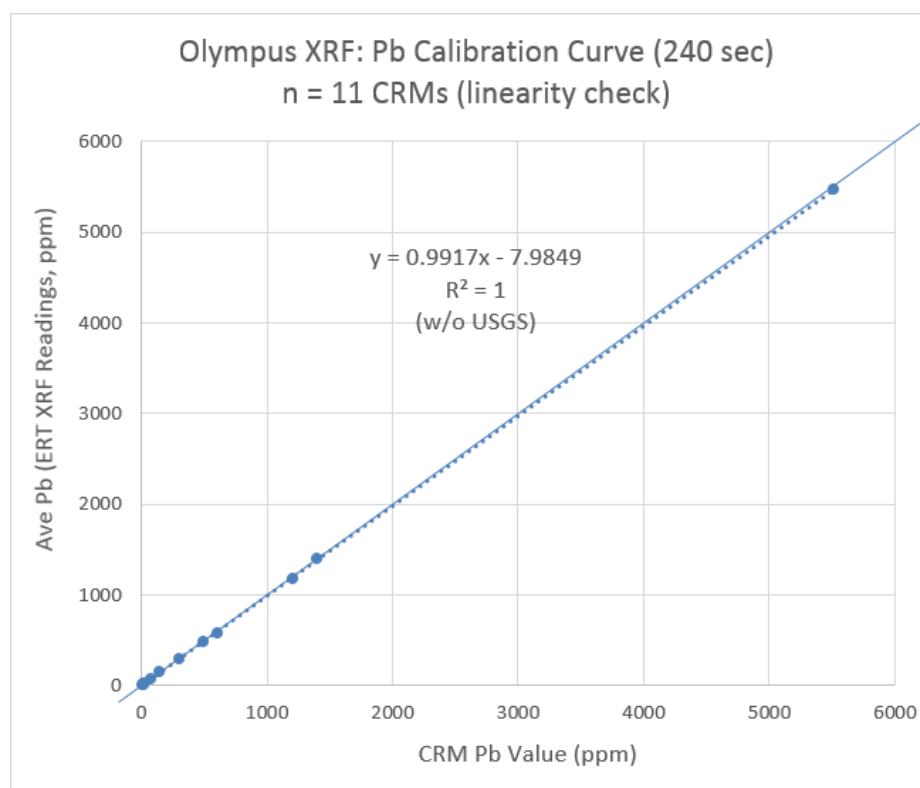
Olympus instrument limit of detection (LOD) for Pb:

- 3.3 ppm (determined as 3 x signal/noise ratio, on a CRM with 11 ppm Pb concentration, and a 240-sec read time)
- 4.2 ppm (determined as 3 x signal/noise ratio, on a CRM with 24 ppm Pb concentration, and a 240-sec read time)
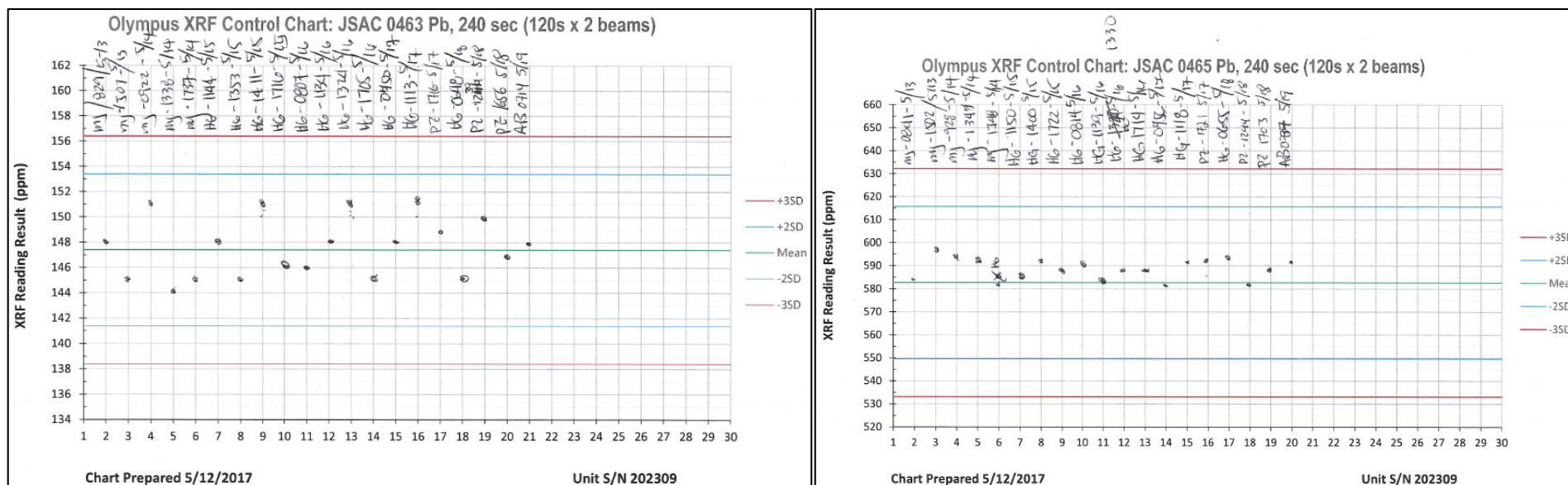
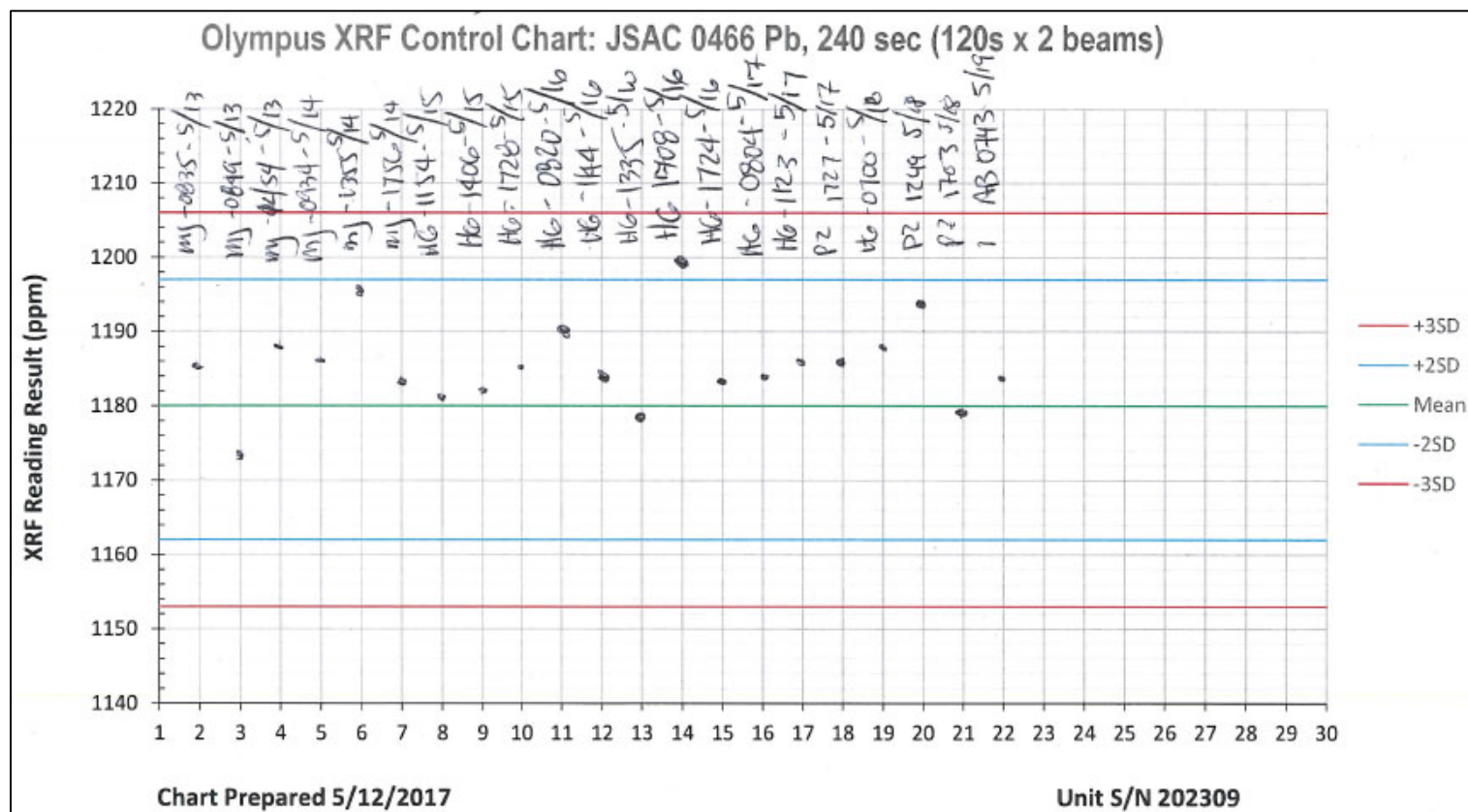Olympus instrument Quantitation Limit (QL) for Pb:

- 14 ppm (determined as the concentration at which the analytical %RSD = ~10 % in a "clean" matrix with a 240-sec read time)

Olympus instrument analytical precision:

- 11.6% RSD (on CRM with 11 ppm Pb and a 240-sec read time)
- 1.5% RSD (on CRM with 300 ppm Pb and a 240-sec read time)

Recent Olympus control charts for 3 concentrations levels for Pb are shown below

Olympus XRF Control Chart: JSAC 0466 Pb, 240 sec (120s x 2 beams)

Chart Prepared 5/12/2017   Unit S/N 202309

Olympus LCS performance/control chart summaries (May 2017):

- JSAC 0463 (Low-level Pb) mean: 147 ppm (cert: 151.6 ppm = 97%); ±2 SD range: 141-153 ppm (±4.1%)
- JSAC 0465 (Mid-level Pb) mean: 583 ppm (cert: 612.4 ppm = 95%); ±2 SD range: 550-615 ppm (±5.7%)
- JSAC 0466 (High-level Pb) mean: 1180 ppm (cert: 1214 ppm = 97%); ±2 SD range: 1160-1197 ppm (±1.6%)

Attachment E: Sample Labeling and Identification

In the sample logbooks for LSA and EBA,
- Record the GPS reading for the center of each SU (including each member of a couplet, which are placed so that there is 1-2 ft. between their adjacent edges), and
- Record the SU configuration as the area and number of increments.
- DUs are GPS at their corners. Record the number of increments per field sample, as well as the number of replicate field samples within a DU.

Proposed Sample Labeling Schema for LSA

LSA SUs
Test SUs for optimizing configuration (all have couplets):
- LSA-Test1-SUXY   where X = 1, 2, 3… for numerical order of collection; and Y = A or B for the couplet member for testing the default configuration (2x2 sq.ft/9 inc)
    o Example: LSA-Test1-SU1A and LSA-Test1-SU1B
- LSA-Test2-SUXY   as above, but for testing the second configuration (3x3 sq.ft/12 inc), if needed
- LSA-Test3-SUXY   as above, but for testing the third configuration (4x4 sq.ft/16 inc), if needed

Delineation SUs along transects (some have couplets):
- For transect SU samples <u>without a couplet</u>: LSA-SUTW,X   where T = "transect", W = transect number (the transect numbering may follow the labeling given in the figure below or the numerical order of sampling as work progresses in the field); X = 1, 2, 3… for numerical order of collection along that transect
    o Example, for the first and second SU samples collected along transect #16: LSA-SUT16,1 and LSA-SU16,2
- For transect SU samples <u>with a couplet</u>: LSA-SUTW,XY   as above, except that Y = A or B for the couplet member
    o Above SU example but with the second transect location having a couplet: LSA-SUT16,2A and LSA-SUT16,2B

Fill-in SUs between transects (some may have couplets):
- For fill-in SU samples <u>without a couplet</u>: LSA-SUFW-W,X   where F = "fill-in", W-W = the 2 transects between which the SU is located; X = 1, 2, 3… for numerical order of collection between those transects
- For fill-in SU samples <u>with a couplet</u>: LSA-SUFW-W,XCY   as above, except that Y = A or B for the couplet member
   o Example of the first fill-in SU couplet between transects 8 and 9: LSA-SUF8-9,1A and LSA-SUF8-9,1B
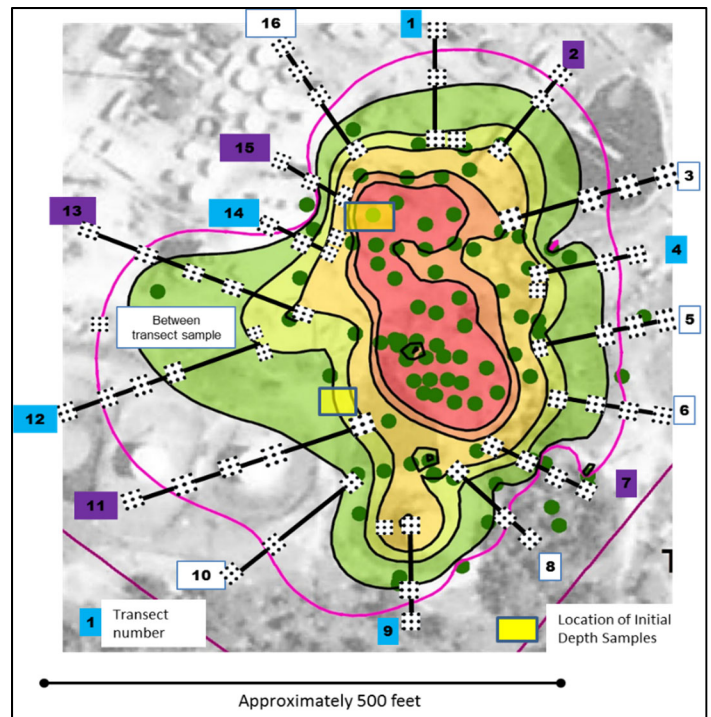
LSA DUs

In the sample logbook for LSA,

- Record the GPS readings for the corners of the DU, and
- Record the DU area and number of increments.

Boundary confirmation DUs:

- For initial placement and sampling of a border segment DU having replicate field samples: LSA-DUTW-W,1RY where T = "transect"; W-W = the 2 transects that the border DU bridges; 1 = the first DU between those 2 transects; R = "replicate"; and Y = A, B or C for the triplicate member
    o Example, an LSA DU that bridges transects 14 and 15, with 3 replicates, would have these sample IDs:
        ▪ LSA-DUT14-15,1RA, LSA-DUT14-15,1RB, and LSA-DUT14-15,1RC
- For initial placement and sampling of a DU NOT having replicate field samples: LSA-DUTW-W,1   as above except no R or Y designations
- For placement and sampling of a shifted or step-out DU having replicate field samples: LSA-DUTW-W,2RY where T = "transect"; W-W = the 2 transects that the border DU bridges; 2 = the first step-out DU between those 2 transects; R = "replicate"; and Y = A, B or C for the triplicate member
- For placement and sampling of a step-out DU NOT having replicate field samples: LSA-DUTW-W,2   as above except no R or Y designations
    o Example continuing the previous example: if LSA-DU14-15 needed to be stepped out because the first bridging DU was >200 ppm, and if it had no replicates, the DU would have this sample ID: LSA-DU14-15,2


===
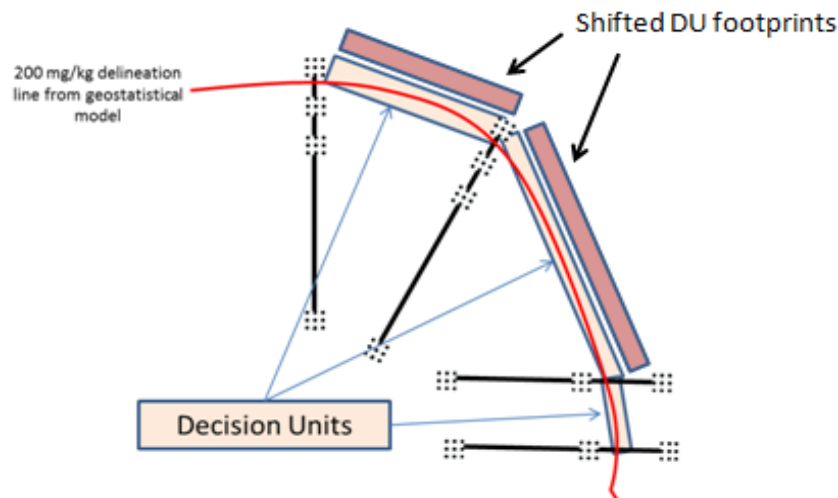
(EBA is below)

EBA SUs

## Proposed Sample Labeling Schema for EBA

- For SUs that <u>search for sources</u> or hot spots around the structures: EBA-SUSX   where the S after SU = "source"; X = the numerical order of sampling the flagged potential source area (PSA) locations
  - Example of the fourth PSA sampled in the EBA: EBA-SUS4

- For SUs that <u>characterize a detected hot spot</u> found by a EBA-SUSX sample: EBA-SUSX-Y   as above, with the Y = the numerical order of collecting additional SUs around SUSX
  - Example of the fourth PSA SU sampled in the EBA, which was >200 ppm and needed to be delineated by surrounding with SUs (as shown in the figure): EBA-SUS4-1, EBA-SUS4-2, EBA-SUS4-3, EBA-SUS4-4, and EBA-SUS4-5

- Hot spot delineation that is guided by *in situ* <u>XRF readings</u> will use the following:
  - XRF operator: The operator will enter the SU identifier (ex: EBA-SUS4) into the instrument. It is not necessary for the XRF operator to add an additional identifier to the individual readings within the same SU. The instrument reading number will be the identifier that links the instrument data download to the XRF logbook record.
  - Field logbook: The recorder for the field crew will make a sketch of the SU and its environs in the field logbook, including GPS coordinates.
    - Reading results will be recorded into the sketch in the field logbook, along with the order number of the location being tested (such as Loc #1, #2, etc.)
    - Replicate shots in the same location to control for particle effects will be designated with A, B, C, etc. If only a <u>single</u> reading is taken in a location within the SU, designate this with an S after the Loc#.
      - SUs or locations that appear to show little variability do not need frequent replicate readings in the same location.
  - XRF logbook: The recorder will enter the SU identifier (e.g., EBA-SUS4), the Loc #, and the replicate letter, plus the instrument reading number, the clock time the reading was taken, the acquisition time of the reading (e.g., 15 sec), the Pb result & instrument error (as 1 SD).
  - See the figure below for an example of field and XRF logbook entries for an SU
  - *In situ* field results are not entered into an RTeX form because they are used qualitatively to understand contaminant patterns within an SU, or to chase contamination that appears to have migrated.

- For <u>transect</u> SUs in the EBA (if it is discovered they are needed): EBA-SUTW,X   where T = "transect", W = transect number in order of sampling, and X = numerical order of SUs collected along that transect
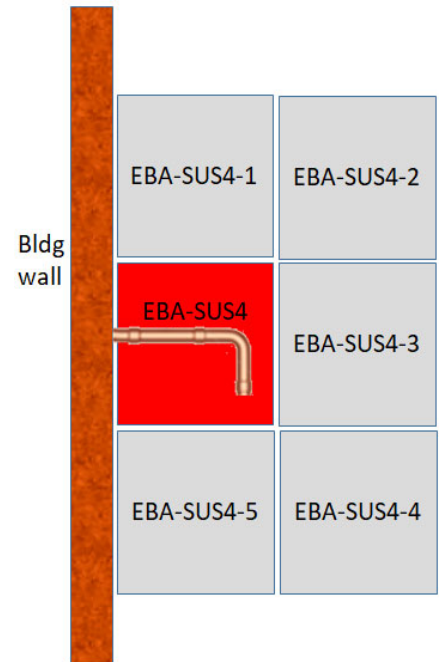
In XRF Logbook

In Field Logbook

EBA-SUS4

SU ID	Rdg#(Inst)	Time	AcqTime	Rdg ID	Pb (err)
EBA-SUS4	358	1430	15 sec	Loc#1A	156 (4.3)
	359	1430	15 sec	Loc#1B	184 (4.7)
	360	1431	15 sec	Loc#1C	175 (4.6)
	361	1431	15 sec	Loc#2A	190 (4.3)
	362	1431	15 sec	Loc#2B	210 (4.4)
	363	1432	15 sec	Loc#3S	195 (4.4)
	364	1432	15 sec	Loc#4A	650 (6.7)
	365	1433	15 sec	Loc#4B	484 (5.5)
	366	1433	15 sec	Loc#4C	575 (6.0)

Loc #1
156
184
175

Loc #2
190
210

Loc #3
195

Loc #4
650
484
575
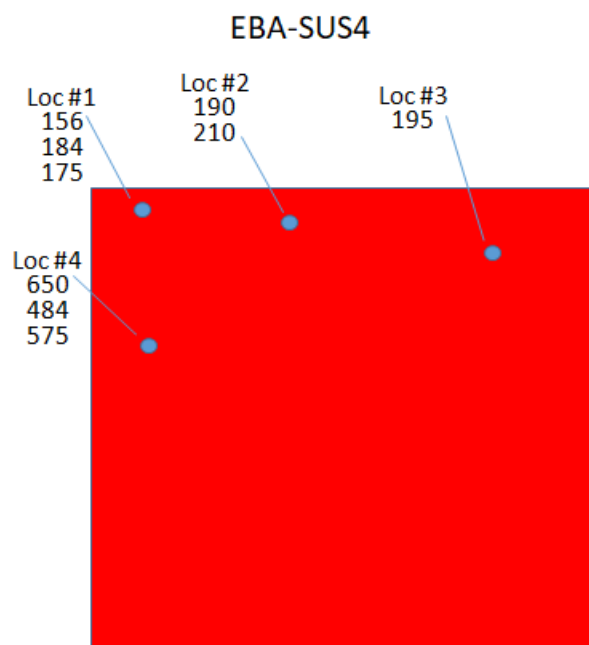
EBA DUs
- For DUs around the structures: EBA-DUX where X = numerical order of DUs being collected.
- Triplicate DUs have A, B or C attached.
- The first five DUs sampled in the EBA are expected to have these sample IDs (Refer to Attachment B, Section :
  - EBA-DU1A, EBA-DU1B, and EBA-DU1C
  - EBA-DU2A, EBA-DU2B, and EBA-DU2C
  - EBA-DU3A, EBA-DU3B, and EBA-DU3C
  - EBA-DU4
  - EBA-DU5